

University Of Alberta



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TEACHER'S LABORATOR GUIDE

for
Chemistry:
Experiments
and
Principles

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MacNab
Davis
Haenisch
O'Connor

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1	H
1.008	

3	4
Li	Be
6.94	9.01

11	12
Na	Mg
23.0	24.3

19	20
K	Ca
39.1	40.1

37	38
Rb	Sr
85.5	87.6

55	56
Cs	Ba
132.9	137.3

87	88
Fr	Ra
(223)	(226)

2	He
4.00	

9	10
F	Ne
19.0	20.2

17	18
Cl	Ar
35.5	39.9

35	36
Br	Kr
79.9	83.8

53	54
I	Xe
126.9	131.3

85	86
At	Rn
(210)	(222)

PERIODIC TABLE

5	6	7	8	9
B	C	N	O	F
10.8	12.01	14.01	16.00	19.0

13	14	15	16	17
Al	Si	P	S	Cl
27.0	28.1	31.0	32.1	35.5

31	32	33	34	35
Ga	Ge	As	Se	Br
69.7	72.6	74.9	79.0	79.9

49	50	51	52	53
In	Sn	Sb	Te	I
114.8	118.7	121.8	127.6	126.9

81	82	83	84	85
Tl	Pb	Bi	Po	At
204.4	207.2	209.0	(210)	(210)

30	29	28	27	26	25	24	23	22	21
Zn	Cu	Ni	Co	Fe	Mn	Cr	V	Ti	Sc
65.4	63.5	58.7	58.9	55.8	54.9	52.0	50.9	47.9	45.0

48	47	46	45	44	43	42	41	40	39
Cd	Ag	Pd	Rh	Ru	Tc	Mo	Nb	Zr	Y
112.4	107.9	106.4	102.9	101.1	(99)	95.9	92.9	91.2	88.9

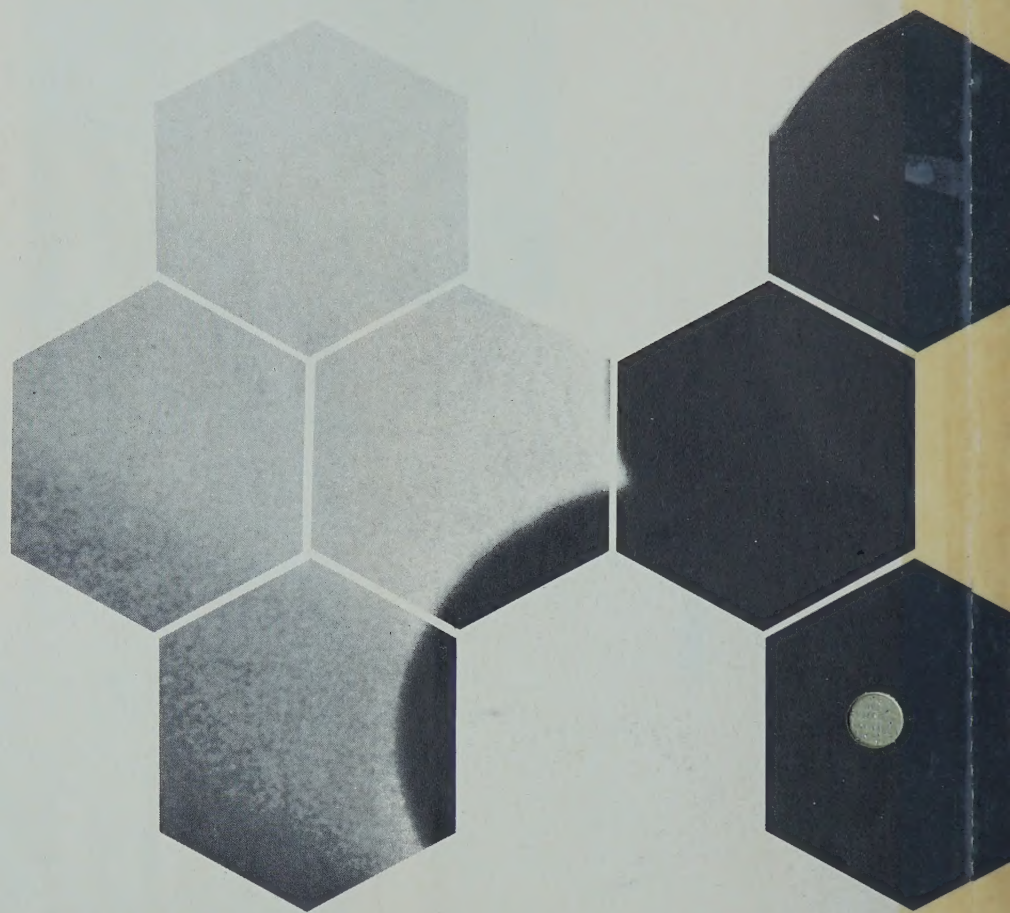
80	79	78	77	76	75	74	73	72	71
Hg	Au	Pt	Ir	Os	Re	W	Ta	Hf	La-Lu
200.6	197.0	195.1	192.2	190.2	186.2	183.9	180.9	178.5	89-103

100	99	98	97	96	95	94	93	92	91
Fm	Es	Cf	Bk	Cm	Am	Pu	Np	U	Pa
(253)	(254)	(249)	(247)	(248)	(243)	(242)	(237)	238.0	(231)

101	102	103
Md	No	Lw
(256)	(254)	(257)

57	58	59	60	61	62	63	64	65	66	67	68	69	70	71
La	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
138.9	140.1	140.9	144.2	(145)	150.4	152.0	157.2	158.9	162.5	164.9	167.3	168.9	173.0	175.0

89	90	91	92	93	94	95	96	97	98	99	100	101	102	103
Ac	Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lw
(227)	232.0	(231)	238.0	(237)	(242)	(243)	(248)	(247)	(249)	(254)	(253)	(256)	(254)	(257)





TEACHER'S LABORATORY GUIDE

for Chemistry:
Experiments
and Principles

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Cover design by Harold Pattek

Illustrations by PIP, Inc. Studio
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Printed in the United States of America

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Introductory Guide to the Laboratory Work

The treatment of laboratory work differs from that employed in many conventional courses. The student often investigates properties of matter in the laboratory before they are brought up in class. He gathers some facts—that is, gets an experimental basis for understanding concepts—which you and the class can discuss the next day. This technique is superior to sending the student into the lab to verify generalizations made by the Textbook or the teacher.

Most experiments should receive a short prelab discussion on problems of experimental technique and a postlab session devoted to the systematic development of relevant principles. The experiments are described for an ordinary scale of operations. Semimicro scale can be used by making certain obvious changes in a few experiments. Since a reduction in sample size might increase errors in weighing, careful consideration should be given to this possibility before reducing the scale of experiments which require weighing.

The following general comments about laboratory matters apply to all experiments. These remarks, plus the individual guides, are to help you plan the laboratory work. The best student results will be obtained when you have prepared well ahead of time.

NOTEBOOKS

Keeping a permanent record of experimental data is an important part of scientific training. There are a number of ways this can be accomplished. Some teachers prefer to have the students prepare a carbon copy of their lab observations to be turned in at the end of the lab session. This procedure forces the students to make a record, but has the disadvantage of overburdening the teacher with stacks of papers to process. An alternative method is to have the students keep a permanent record in a bound laboratory notebook. The teacher can supply dittoed report forms which the student prepares in duplicate. The original can be handed in and the copy can be pasted into the record book. Generally these reports can be one-half page or less, which simplifies processing.

Answers to questions in *Processing the Data* should either be written in the permanent record or prepared in duplicate so that the student has a copy for reference when the experiment is discussed.

Whichever method is used, constant encouragement is needed to help the students develop this important aspect of scientific investigation. Neatness should be fostered but not to the degree that it interferes with the student's use of the notebook as an original record. Encourage thorough preparation of data tables and familiarity with the procedure before students come to the laboratory. It is useful to occasionally forbid use of Lab Manuals in the lab. This forces the students to prepare in advance and reduces wear on manuals.

USE OF THE LABORATORY

In general, it has been assumed that the class is held in a combined laboratory-lecture room. A 50-minute period is used as a standard. Longer periods are desirable, and can be used to

advantage in almost every experiment, permitting additional work and extensions of the experiments.

CHEMICAL AND EQUIPMENT LISTS; FILM SOURCES

Complete lists of the chemicals and equipment required will be found at the back of this Guide. Also located at the back of the Guide is a list of sources from which the films may be ordered.

Teacher's Guide to Individual Experiments

The Laboratory Guide is arranged by textbook chapters. A short summary explains the main motif of the experiments. A table shows the suggested order of text sections, experiments, and demonstrations. It also serves as an index for that chapter of the Guide. An example is given below to show the system used. The "Comment" column is not included in the actual tables.

Schedule of Experimental Work

TEXT SECTION	EXPERIMENTAL WORK	FOR DETAILS SEE PAGE	COMMENTS
	Demo. 2	18	Demo. 2 and Expt. 5 should precede
	Expt. 5	19	Section 2-1. Demos. 3-6 come
2-1	Demo. 3 to 6	25	<i>during</i> Section 2-1.
2-2 to 2-5			
2-6	Demo. 7	26	The details of Demo. 7 are on Lab
2-7 to 2-9			Guide p. 26.
	Demo. 8	30	
	Expt. 6 } use of both	31	
	Expt. 7 } optional	34	
	Expt. 8	37	
2-10			

For each experiment the same basic outline is used, although some parts do not occur in every experiment. The demonstrations are presented in various ways as they differ in preparation needed. They are all numbered for reference.

PURPOSE AND SUMMARY

The purpose is stated from the teacher's point of view. Of great importance is the need for the student to plan for his experiments. Detailed instructions have been included for lab experiments to make administration easier for you. Impress the student with your expectation that he will study the procedure before class and be ready to begin work when he comes into the lab. Students should be encouraged to evaluate the procedures given and to suggest extensions of the experiments. They should be encouraged to follow up on these whenever time and interest permit.

The summary is quite short and intended to remind you what the student is doing. It is not a summary for class presentation.

TIMING

A statement suggests where in the textbook the experiment fits best. An estimate of the amount of time required in the laboratory is given along with suggested stopping points if they are needed. Some experiments require portions of several periods (usually to make use of over-night drying). Therefore, this part of the guide will help you plan for the best use of time.

MATERIAL NEEDED

Equipment and supplies are listed per student or per pair, except as indicated. Some teachers prefer to have no equipment in the desk at the beginning of the term, and then to provide it as needed. This has the distinct advantage of confronting the student with fewer strange pieces of equipment at one time. Others set out all equipment for each experiment. This method helps reduce losses by breakage.

A list of chemicals is included and directions are given for preparing solutions.

Where the word *concentrated* appears after the name of an acid or ammonia, it means the solution from a freshly opened bottle of commercially available reagent. The molar concentrations of these reagents are shown in the table.

Average Properties	HCl	HNO ₃	H ₂ SO ₄	NH ₃ solution	CH ₃ COOH
Specific gravity	1.19	1.42	1.84	0.90	1.06
% Present	37	70	96	29	100
Grams active ingredient/ml	0.426	0.985	1.76	0.527	1.055
Concentration, <i>M</i>	11.7	15.6	18.0	15.1	17.6

It is, of course, necessary for the teacher to make preparations well in advance. To aid in this, experiments for which lengthy or long-range preparation is needed are indicated, in advance, in the *Schedule and Related Material* section.

Convenience in dispensing chemicals and equipment is of great importance to avoid wasted time and boredom from waiting. The best practice is to arrange for enough containers such that students do not consume precious time from an already short period.

LABORATORY HINTS

Substitutions for apparatus, solutions to special problems, alternate ways to proceed and points for teacher background are included in this section.

PRELAB DISCUSSION

This briefing should be short and devoted largely to experimental or manipulative details. Do not reveal any "element of discovery." In general, the laboratory manual contains sufficient description of purpose.

Make sure your announcements are clear. Try to avoid interrupting the lab work later with further remarks. It is difficult to get attention.

PRECAUTIONS

The phrase "Observe the usual lab precautions" is given to suggest the constant dangers of mishandling even common chemicals and equipment. More specific reminders are also frequently listed.

POSTLAB DISCUSSION

This is the place to make sure the students understand the experiment and the regularities it demonstrates. The questions help draw this out. Use a small part of this period to comment on the form of reports.

SAMPLE DATA

The sample data often includes actual student experience. It gives an idea of the spread of results to be expected and suggests ways to present the class data so each student can see all of it. Where appropriate, suggested report forms have been included. You will probably find the material easier to check in summary form than if you were to go over the entire lab report. It is of great importance to discuss the results obtained by the entire class, since students normally do each experiment only once and thus do not have a means of evaluating precision. Whenever possible, results should be posted on a chart of some sort (specific suggestions are given) and the regularity discussed.

One way to use this material is for you or the students to determine the range of the middle 60% or 70% of the results. This range represents (roughly) plus and minus one standard deviation from the mean. Leaving out the highest and lowest 15–20% eliminates results based on gross errors.

RESULTS OF PROCESSING DATA

This section provides samples of the calculations to be made and answers to questions. These have been answered in your vocabulary, in general, although "expected" answers from students have been included in many cases.

ADDITIONAL INVESTIGATIONS

At the end of many experiments is a brief suggestion for further work. This is for *only* the most able students. No procedure is indicated, and certainly no student should be encouraged to continue unless he has some idea of how to go on. For some experiments, suggestions are included.

QUESTIONS TO WONDER ABOUT

These questions are to stimulate thinking but are not to be assigned as written work. They vary in scope and difficulty, and some have no specific answers. Discussion of some of these questions will be included soon; and others, later in the Textbook.

Chemistry: An Experimental Science



The first chapter is intended to stress observation and organization with the goal of finding regularities. We assume the student has little experience in the laboratory. The four experiments are very simple; weighing and measuring volume or length are the only operations required.

Schedule of Experimental Work

TEXT SECTION	EXPERIMENTAL WORK	FOR DETAILS SEE PAGE
	Expt. 1	2
1-1, 1-2		
	Expt. 2	4
1-3		
	Expt. 3	6
1-4, 1-5	Demo. 1	11
1-6, 1-7		
	Expt. 4	12
1-8 to 1-10		

Experiment 1

Scientific Observation and Description

PURPOSE AND SUMMARY

To emphasize the importance and value of careful observations and to illustrate how many observations can be made on a seemingly simple system. The student observes a burning candle and describes what he observes.

PRELAB DISCUSSION

Give a very short introduction. This experiment illustrates the philosophy of this course. This is not a course in which the student listens while the teacher talks about science; rather it is one in which the student participates in science. He becomes actively involved in doing what scientists do. He will find out what science is, primarily as a result of his own experiences.

Do not give him suggestions as to what to observe, but be certain he knows the assignment: to make and record (original plus one carbon) all his observations on a burning candle.

Do not hand out lab manuals until after the experiment. Have the student start this course by doing an experiment, relying on his own resources. This procedure is safe for this very simple experiment.

TIMING

Do this experiment on the first day, before giving anything except the briefest of introductions to the course. Takes about 10–15 minutes in the lab.

MATERIALS NEEDED

per student

candle, mounted on a "drip catcher." See Lab Hints 2 and 3.
matches
metric rulers

PRECAUTIONS

A brief comment about not burning noses or hair would be appropriate.

LAB HINTS

1. Experience shows that this exercise in observation sufficiently engrosses the students that the teacher will be given an excellent opportunity to complete some of the first day's routine. *Laboratory Manuals are needed for directions and are to be given out at the end of the first day.*
2. Be sure candles are not the "dripless" type. Ordinary, inexpensive candles sold under the name "household candles" are satisfactory. A six-inch candle ($\frac{3}{4}$ inch diameter) is fine.

3. Cardboard squares or can lids make good drip catchers. Unless one is provided, the students tend to use the wire gauze with asbestos center, and this causes difficulties later.

POSTLAB DISCUSSION

After the observation period, collect a copy of the student's observations. This is an important step in establishing routine. Make clear to your students that a written description of the burning candle is due the next day. (See the last paragraph in Laboratory Manual *Procedure*.)

Base your discussion of the difference between observation and interpretation upon the "observations" the students make. An observation is simply a record of what is observed. It is devoid of reference to significance, implication, or indirect relation to other observations. An observation coupled with a valid estimate of uncertainty constitutes a "fact," and it is considered to be a statement of a property of nature, unchanging in time.

An interpretation includes reference to significance, implication, or indirect relation to other observations. In essence, interpretation involves an attempt to draw meaning from one or more observations or to establish causal relations.

Operationally the distinction is simple, though students are not used to keeping the two separate in their minds. A few examples will make clear the distinction. Usually the interpretation is most readily exposed by proposing an alternate meaning or relationship that cannot be discounted with the available information. Here are two examples.

1. *Observation:* Jim and Jane are frequently seen together.
Interpretation: Jim and Jane are "going steady."
Possible alternate interpretations: Jim and Jane are brother and sister. They have accidentally become handcuffed together.
2. *Observation:* If you pass your finger through a candle flame, it feels hotter just above the tip of the flame than it does in the middle.
Interpretation: Heat rises.
Possible alternate interpretation: Heat is being produced at the tip of the flame, and some is being consumed at the middle (for example, the heat might be used to produce the light emitted by the flame).

Notice that an interpretation "heat rises" might be defended by some student on the basis that it is known from other data. If so, ask him what data he has in mind, and again point out that the remark "heat rises" is an interpretation readily distinguished from the new observation he proposes. Draw attention to the fact that classifying the statement "heat rises" as an interpretation is not a *denial* of the statement, but that it is merely a distinction between what is observed (which does not change) and the significance attributed to the observation (which, experience shows, frequently does change).

Emphasize the difference between qualitative and quantitative observations. This may be brought up during the postlab discussion or it may be delayed until the lab write-up is in. Any observation involving contrasts is, to some extent, quantitative.

For example, the statement "Light is emitted by a candle" is qualitative, whereas the statement "The candle flame appears bright in a normally lighted room" is quantitative, since it provides some basis for judging *how much* light is emitted by the candle. Still more quantitative (and hence more useful) would be the statement: "The candle flame emits about the same amount of light as one square centimeter of iron heated to $700 \pm 50^\circ\text{C}$."

When the textbooks are passed out on the second day, the student should be directed to read the observations on a burning candle in Appendix 1. When he compares his observations with those of a professional scientist, the student may be shocked by the number of things he failed to report. This is intended; exploit it. Point out that the professional scientist has years of training and experience behind his ability to observe carefully. It is an important aim of this course to train the student to observe more carefully and more fully.

The student's initial observations are the recording of simple sensory responses. As he becomes acquainted with more sophisticated instruments to augment his senses, the precision with which he is able to make an observation will increase. The accuracy of the observation, however, depends upon the extent to which he actually observes (or measures) what he intends to observe. The degree of uncertainty in an observation depends on both precision and accuracy. Usually the uncertainty due to precision can be learned merely by repeating the experiment many times. The uncertainty due to accuracy is difficult to ascertain. The same quantity must be measured by several methods. When only the uncertainty in precision is known, it is usually called "the uncertainty," though it is understood that accuracy is untested.

Experiment 2

Observing Evidence of Interaction

PURPOSE AND SUMMARY

To provide additional experience in making careful observations using an unfamiliar system and to raise some rather sophisticated questions which will be considered later in the course.

The student prepares a solution of cupric chloride and observes what happens when aluminum foil is allowed to interact with the solution.

TIMING

Following Experiment 1 and after reading Section 1-2 in the Textbook. About 15–20 minutes are required. The reaction takes about 10 minutes.

MATERIALS NEEDED

per student

- 1 beaker, 100 ml
- 1 thermometer, Celsius, -20 to 150° range
- 1 piece aluminum foil, 10 cm $\times$ 20 cm
- 2 level teaspoonsfuls, cupric chloride dihydrate, $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$

per class

several wide-mouthed reagent bottles of solid cupric chloride and plastic teaspoons

LAB HINTS

None for the students. You will be interested in the following.

Cupric chloride dissolves in water to form a solution which is acidic. The *pH* of a 1 *M* solution is about 3. When an excess of aluminum reacts with a cupric chloride solution, there are two principal reactions. Aluminum reacts with hydrogen ions and is oxidized while hydrogen ions are reduced. At the same time cupric ions are reduced as additional aluminum is oxidized. These reactions are represented by the following equations:



If too little aluminum is used in the reaction, a large amount of white cuprous chloride is produced in a side reaction.

PRELAB DISCUSSION

Review briefly common errors made by students in making observations of a burning candle system. Have the students recall the difference between an observation and an interpretation. Discuss ways of making quantitative observations. Make sure the students know what is expected and how they are to proceed. Encourage them to make their record as comprehensive as they can (original plus one carbon).

PRECAUTIONS

Now is a good time to start stressing that the student should *never* taste chemicals unless he is told it is all right by the teacher. Copper chloride is toxic if taken internally. Remind the students that they should always wash their hands after working in the laboratory. The reaction vessel may get quite hot. Wear safety glasses.

POSTLAB DISCUSSION

After the experiment, collect a copy of each student's observations and questions. Use these observations and questions as a basis for the postlab discussion. Do not attempt, at this time, to have the students understand the chemistry involved in the reaction. Rather, help them distinguish between valid observation and interpretation using their data as examples. You may find the observations given under the *Sample Data* section helpful during the postlab discussion.

SAMPLE DATA

Some Observations Made on the Aluminum-Cupric Chloride Reaction

The temperature of the water was 24.5°C before the blue crystals were added. The blue crystals seemed to turn yellowish-green when first put into water. The solution above the crystals became light blue but remained yellowish-green near the crystals in the bottom of the beaker. Stirring the solution caused the crystals to dissolve faster, making the liquid a deeper blue. The temperature of the liquid increased as the crystals dissolved, warming to about 27°C.

Soon after the aluminum foil was placed in the blue solution, bubbles began to form on the foil. The bubbling became more vigorous as time went on. A reddish-brown, spongy looking substance formed on the aluminum foil and fell to the bottom of the beaker. As the reaction continued, part of the aluminum foil disappeared. More of the reddish-brown material formed. The color of the liquid gradually faded. As the color faded the solution turned milky, then reddish-brown, and finally became colorless. Bubbling continued for about 10 minutes. The temperature of the system increased rapidly to about 90°C maximum and then decreased gradually to room temperature. Some of the aluminum foil remained unchanged after the reaction stopped.

ADDITIONAL INVESTIGATIONS

Some interested students may wish to try to identify the reddish-brown substance produced in the experiment. If the residue is washed and blotted using paper towels, it can be pressed or hammered so that it has a metallic luster and can be identified as being like copper. Some students may want to melt the copper. This can be done using a charcoal block, a propane torch, and some borax as a flux. If the copper and borax are heated enough to melt the copper, they will draw together into a bead. If the hot bead of borax and copper is dropped into some dilute hydrochloric acid, the flux is removed and a shiny piece of copper metal results.

Experiment 3

Searching for a Regularity

PURPOSE AND SUMMARY

To permit the student to discover that the relation between mass and volume is constant for a given substance. The student also gains experience in using balances, graduated cylinders, and scales.

Each student will determine the mass and volume of at least two specimens. He plots data from about 40 specimens. See Lab Hints 1 and 2 for a data sheet and a method of disseminating data.

TIMING

Between Sections 1-3 and 1-4. About 20 minutes needed.

MATERIALS NEEDED**per student**

1 graduated cylinder, 25 ml or as required by size of specimen. Needed only if volume is determined by displacement.

2 specimens. See Lab Hint 4.

per class

balances, centigram

calipers, one pair for each 2 students. Not needed if volume determined by displacement.

LAB HINTS

1. A data sheet form is shown below. If the student keeps a carbon copy, you have the original which can be used for compiling data. Each student needs about 40 pairs of data, half of which are for one substance.

Name _____ Date _____ Period _____

EXPERIMENT 3 Searching for a Regularity

Balance Number _____

Data

Specimen number		
Mass		
Volume of water and metal		
Volume of water		

If volume is to be determined using linear measurements, space will be needed for the required dimensions.

2. Data obtained by the class can be tabulated, as below, and duplicated for the student.

Mass-volume data from 54 different specimens (iron or aluminum)

Mass (g)	Volume (ml)	Mass (g)	Volume (ml)
64.0	8.0	49.5	6.3
55.1	7.5	54.6	8.2
25.5	9.2	21.4	7.1
31.6	4.7	29.8	9.8
45.7	5.8	44.9	5.4
18.7	6.5	49.1	7.3
20.0	8.1	38.6	5.8
38.5	5.2	17.0	8.4
47.5	6.7	10.2	5.8
35.0	7.2	11.8	7.4
27.5	8.6	16.3	5.2
55.5	6.7	20.0	6.1
18.7	6.7	52.5	6.1
15.7	5.6	56.2	7.1
66.4	8.9	25.6	8.7
13.8	6.6	46.2	5.1
9.0	4.3	20.6	5.7
60.0	8.6	56.9	7.9
42.6	6.3	14.7	4.5
25.5	7.6	64.7	8.6
40.3	4.9	67.1	8.4
62.5	7.7	66.1	8.3
19.0	9.5	44.3	6.6
22.5	7.9	58.1	8.2
22.4	8.8	15.0	4.6
69.3	9.0	17.6	7.3
62.2	7.4	16.5	7.4

3. The student can gain experience in using the balance if a set of specimens is available for him to weigh before and after class. An answer key should be posted so he can determine if he is weighing correctly.
4. Specimens of two different substances should be available. Iron and aluminum are excellent. Their densities (7.7 and 2.7 g/ml) give two well-separated lines on the graph. Some sources are:
- scrap rod or bar stock from the school shop
 - a local machine shop or hardware store
 - a science equipment supplier
- Specimens should vary in size and be numbered uniquely. Volumes between 3 and 9 ml are satisfactory.

PRELAB DISCUSSION

Discuss the method you decide to use for determining volume. Give instructions in the use of your type of balance. See Lab Hint 3. Emphasize the necessity of keeping the data for each specimen together. Your students might require a quick review of graphing. Do not use the word "density" now. Save it for the *Postlab Discussion*.

PRECAUTIONS

Exercise normal lab precautions.

POSTLAB DISCUSSION

Most students will have found the points they plotted clustered around two straight lines. Many will suggest in their answers to Question 3 that this is caused by the specimens being made of one of two different substances. Elaborate this regularity. For each line and for each substance, mass/volume (M/V) is a unique constant. This uniqueness makes this regularity useful for identifying substances without consuming part of them or damaging them. Also this regularity enables us to determine the volume occupied by a given mass or vice versa. Such widely used regularities are usually named. This one is called *density*. This is a good place to point out the reasonableness of science. Density is defined as M/V , not V/M (an argument can also be developed for not using $M + V$, $M - V$, M^V or V^M). The M/V ratio gives a high value to substances we think of as being "heavy." That is, objects with a large mass and small volume have a high density.

Point out to students that terms containing two units are not new to them. A salary given in dollars alone means little; dollars/week is meaningful. Saying something is heavy does not say much. A cupful of mercury is heavy, but you could lift it (7 lb). A large piece of Styrofoam on the other hand is light. A huge $6' \times 6' \times 6'$ cube weighs about 100 lb. You would have a hard time lifting this light thing! When in doubt, give mass/volume rather than mass alone.

There will usually be some "stray" points on the graph that bear discussion and elimination. Their atypical values are caused by blunders rather than lack of precision in the instruments used.

SAMPLE DATA

Given on page 8.

RESULTS OF PROCESSING DATA

- 1) Obtain mass and volume data from other members of your class. Be careful to keep the pairs of data together.

See Lab Hint 2.

- 2) Plot these data on a graph. Record mass along the labeled vertical axis and volume along the labeled horizontal axis. Give the graph a title.

See Figure 1.

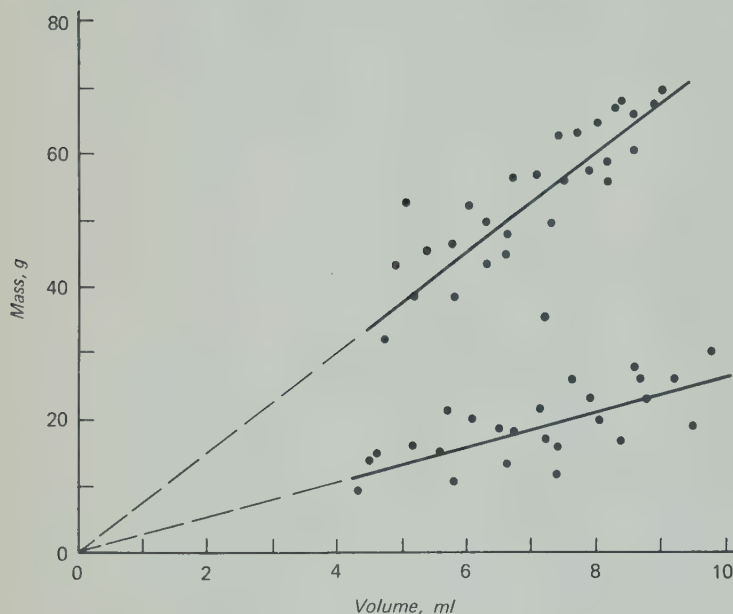


Figure 1
Mass-volume data for specimens of two metals.

- 3) State the regularity, and propose an explanation for what you observed on your graph.

The points cluster around two lines. The separation of points into two groups might be caused by some specimens being made of one metal and others of another metal. A piece of one would have a certain mass. A same sized piece of a different metal might well have a different mass.

- 4) Should each line go through the point (0, 0) ?

Yes, each line should go through the point (0, 0). If the volume of a specimen is zero, its mass would also be zero.

- 5) The introduction to this experiment contains two methods for determining the volume of solids. Which method would give the more precise answer if used on a regular solid? Explain,

The answer depends on the instruments available. Using an ordinary 25 ml graduated cylinder, the volume of a specimen can be determined ± 0.1 ml at best. Using a ruler, linear dimensions can likely be determined to within ± 0.02 cm. A cube 2.00 ± 0.02 cm on each edge would then be reported as 8.00 ± 0.1 ml. Using a graduated cylinder would give 8.0 ± 0.2 ml. The difference is slight. Point out the use of mathematics does not give precision to measurements. For example, the result of multiplying the number 1.36 by 2.41 is 3.2776. However, if 1.36 and 2.41 are numbers resulting from measurements, then the answer is 3.28. To believe the answer is 3.2776 is to believe you can get a precision of one part per 32776 parts from measurements that have a precision of 1 part per 136 parts and 1 part per 241 parts. Were this true, machine shops could stop buying precision micrometers!

ADDITIONAL INVESTIGATIONS

This experiment can be followed by one in which students determine the density of an unknown. Colorless plastics of different density make good specimens.

DEMONSTRATION 1

PRESSURE INDEPENDENT OF TUBE DIAMETER

In discussing Section 1-5 (Measurement of Gas Pressure) you may find a student who thinks the height of a mercury column maintained by gas pressure "should" be different in different sized tubes. His incorrect logic is that the larger tube has more mercury in it so that the column in it will be lower than that in the small tube. This student has forgotten that pressure is force *per unit area*.

You can show mathematically or visually that the same height will be seen regardless of the tube diameter.

$$\begin{aligned}\text{force up} &= \text{force down} \\ &= (\text{mass of Hg})(\text{acceleration of gravity}) \\ &= (\text{height} \times \text{area})(\text{density})(\text{acceleration of gravity}) \\ \text{pressure} &= \text{force/area} = \text{height} \times \text{density} \times \text{acceleration of gravity}\end{aligned}$$

Thus pressure reflects only height and not area of tube.

The illustration shows an apparatus that demonstrates the independence of tube diameter and pressure.

To make this device, select a quart jar with an airtight lid. Cut holes in the lid to take 6, 10, and 25 mm glass tubes. Fashion the bottom ends of the larger tubes at an angle with a wire gauze. Clamp the tubes in place (off the jar bottom) and fasten a light cardboard dam around the jar top. Pour molten beeswax in the dammed space. To use the apparatus, fill the jar two-thirds full of an appropriate colored solution for example KMnO_4 or $\text{Ni}(\text{NO}_3)_2$.

In use, be sure to control the air flow in order that the liquid levels will change slowly. Rapid change reveals a different rate of rise or descent. This interesting aspect, caused by friction with the wall, should not be allowed to obscure the main issue. Stop blowing before the bottoms of the tubes are exposed. The subsequent rush of air sometimes carries liquid out of the tubes onto hapless demonstrators.

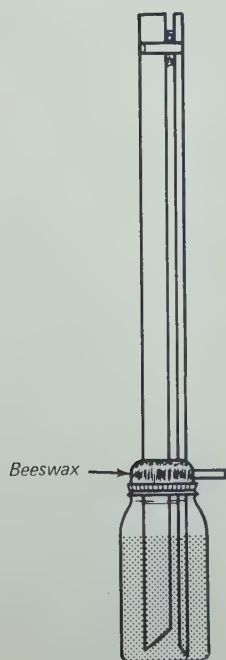


Figure 2

Device to demonstrate that size of tube does not determine liquid level.

Experiment 4

Weighing an Object Immersed in Two Different Fluids

PURPOSE AND SUMMARY

To let the student observe the effect of buoyancy. Mastering this concept now will make Experiment 5 easier. In treating the data, the student sees the volume of an object is more important than mass in determining whether it floats. A chunk of metal sinks; when formed into a boat, it floats.

Each student weighs two objects both in air, and in water. Volume is also determined. This experiment follows very easily after Experiment 3 because the operations required are similar. See Lab Hint 1 for suggested data and calculation sheet.

TIMING

Between Sections 1-7 and 1-8. Allow 30 minutes.

MATERIALS NEEDED

per student

- 1 beaker, 250 ml
- 1 ft thread
- 1 graduated cylinder, 25 ml
- 2 specimens. Pairs matched for volume are needed. See Lab Hint 4.

per class

- balances, centigram
(Need provision for weighing object immersed in water. See Lab Hint 2.)
- calipers, metric. See Lab Hint 3.

LAB HINTS

1. Sample Data Sheet

Name _____ Date _____ Period _____

EXPERIMENT 4 Weighing an Object Immersed in
Two Different Fluids

Balance Number _____

Data

Specimen number		
Mass in air	g	g
Mass in water	g	g
Volume of water + specimen	ml	ml
Volume of water	ml	ml

Calculations

1) Difference between mass in air and in water		
2) Volume of specimen		
3) Mass of water displaced		
4) <u>Mass water displaced</u> Apparent loss in mass of specimen		

Collect the original of each data sheet before students leave lab. The student keeps a carbon copy. Next day give each student the data consolidated from whole class before requiring a write-up.

- Most overhead beam balances have, as part of the pan hanger, hooks from which a specimen can be hung. If the balance lacks facilities for supporting a beaker of water, use a ring stand and ring. Many platform balances can be mounted on a vertical bar and specimens hung from a hook under the pan.

3. The water displacement method for determining volume is simple and recommended. If your graduated cylinders allow volume measurements ± 0.1 ml, then linear measurements will have to be ± 0.04 cm or better to improve the precision.

Volume determined from two readings on graduated cylinder

$$8.0 \pm 0.2 \text{ cm}^3$$

Volume calculated from measurements on a cube

$$(2.00 \pm 0.04)^3 = 8.0 \pm 0.2 \text{ cm}^3$$

or with a good pair of calipers

$$(2.00 \pm 0.01)^3 = 8.00 \pm 0.04 \text{ cm}^3$$

4. Specimens can be obtained as in Lab Hint 4, Experiment 3. Volumes should match (± 0.5 ml) within a pair, and be between 3 and 9 ml. The densities of the specimens within a pair should be quite different. Many substances can be used. Some examples and their densities, g/ml are: lead 11, copper 8.8, cadmium 8.6, brass 8.5, iron 7.7, tin 7.3, zinc 7.1, aluminum 2.7, quartz 2.6, glass 2.5, magnesium 1.7, cellulose acetate 1.3, phenol formaldehyde 1.3–2.0, rubber 1.2, PVC (polyvinylchloride) 1.2–1.6, nylon 1.1.

PRELAB DISCUSSION

The student should be urged to take special care to prevent the object being weighed in water from touching the beaker.

You might choose to say something about the method being used to determine volume.

PRECAUTIONS

Observe normal lab precautions. Spilled water is a minor problem.

POSTLAB DISCUSSION

While correcting the experiments, tabulate the values of the ratios given in answer to question 5. A plot of one set of values is given in Figure 3. Since water was used, the peak at a value of one for the ratio confirms Archimedes' Principle.

The mass of an object apparently depends on the medium in which it is weighed. Yet mass is an invariant property of matter. The dilemma is one of common vs rigorous usage. Mass determinations are usually made in air. The air, however, buoys up the specimens. Its "true" mass is clearly not being determined. Its "apparent mass" is. This misrepresentation is usually not serious—about one gram per liter of matter. Most solids have a mass of over 1000 grams per liter. The misrepresentation is less than 0.1%. "True mass" is determined by weighing in a vacuum.

Discussing the answers to the questions in the write-ups can conclude the *Postlab Discussion*.

SAMPLE DATA AND RESULT OF PROCESSING DATA

- 1) For each object, determine the difference between the apparent mass in air and the apparent mass in water. The reason for using the phrase "apparent mass" rather than "mass" will be clear when this experiment is completed and discussed in class.

	#1	#2
Apparent mass in air	67.8 g	16.2 g
Apparent mass in water	61.8 g	10.0 g
Difference	6.0 g	6.2 g

- 2) Determine the volume of each object.

	#1	#2
Volume of water + specimen	23.5 ml	22.6 ml
Volume of water	17.4 ml	18.7 ml
Difference	6.1 ml	5.9 ml

- 3) Does the mass of an object or its volume have a greater effect on the difference between the apparent mass of an object in air and its apparent mass in water? Explain.

One object had an apparent mass in air over four times that of the other. The volumes were nearly the same. The apparent loss in mass was nearly the same for each object. The volume has the greater effect on the apparent loss in mass.

- 4) Determine the mass of water displaced by each object. One gram of water occupies 1.0 milliliter.

	#1	#2
Mass of water displaced	6.1 g	5.9 g

- 5) Determine for each object, the value of the ratio

	#1	#2
$\frac{\text{mass of water displaced}}{(\text{apparent mass in air}) - (\text{apparent mass in water})}$		
mass of water displaced	6.1 g	5.9 g
(apparent mass in air) - (apparent mass in water)	6.0 g	6.2 g
result of dividing	1.02	0.95

See Figure 3, page 16.

- 6) Express in words the significance of the value found for the ratio in item 5 above.

The mass which an object appears to lose when it is immersed in water is very nearly equal to the mass of the water displaced.

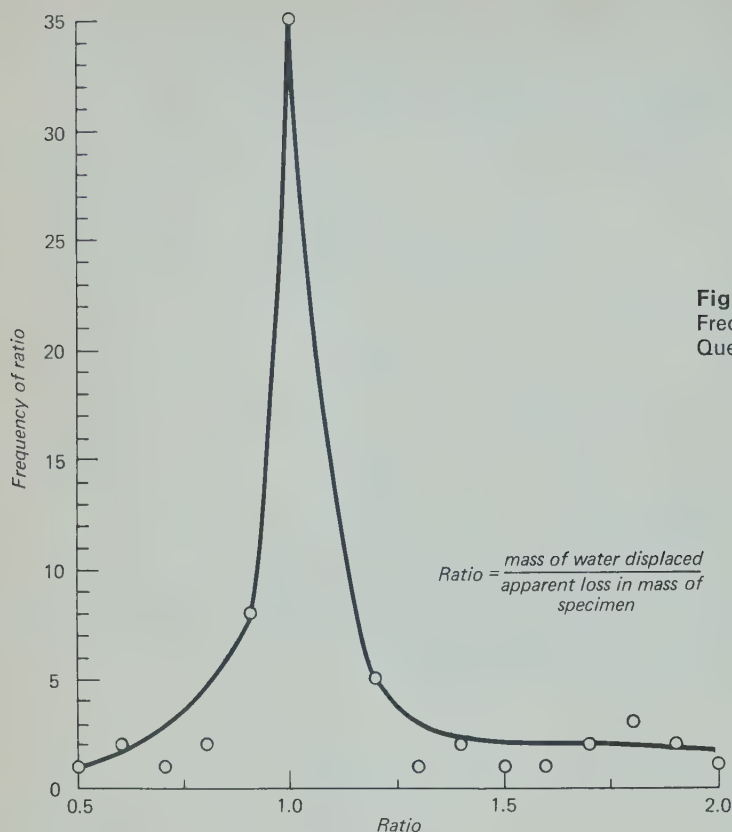


Figure 3
Frequencies of ratio reported in answer to Question 5.

- 7) Will an object have a greater mass in a vacuum or in air? Why?

An object will weigh more in a vacuum than in air. An object weighed in air appears lighter by an amount equal to the mass of the air displaced.

- 8) One liter of air at room temperature and normal atmospheric pressure has a mass of 1.18 g. What would have been the mass of your objects had they been weighed in a vacuum?

Each object would have gained about

$$(6.0 \text{ ml})(0.00118 \text{ g/ml}) = 0.00708 \text{ g}$$

- 9) Why is the adjustment suggested in item 8 above not usually made on weighings made in air?

On the balances used in this experiment, this small difference would not have been detectable. For specimen #1, the difference is $0.01/67.8$ or 0.02%. For specimen #2, the difference is $0.01/16.2$ or 0.06%.

The Atomic Theory



Chapter 2 uses combining volume data to arrive at Avogadro's Hypothesis. The experimental need for the concepts of atoms, molecules, elements, and compounds is seen. The relative mass of molecules is developed experimentally. The experiments themselves are still fairly simple. Study of reactions begins.

Schedule of Experimental Work

TEXT SECTION	EXPERIMENTAL WORK	FOR DETAILS SEE PAGE
	Demo. 2	18
	Expt. 5	19
2-1	Demo. 3 to 6	25
2-2 to 2-5		
2-6	Demo. 7	26
2-7 to 2-9		
	Demo. 8	30
	Expt. 6 } use of both	31
	Expt. 7 } optional	34
	Expt. 8	37
2-10		

DEMONSTRATION 2

THE MASS OF AIR

PURPOSE

To show air has mass and to determine the mass of a liter of air.

TIMING

Immediately prior to Experiment 5. Requires about 10–15 minutes.

MATERIALS NEEDED

balance, centigram

conical flask, 250 ml with stopper as shown in Figure 4

vacuum pump (an aspirator if nothing better is available)

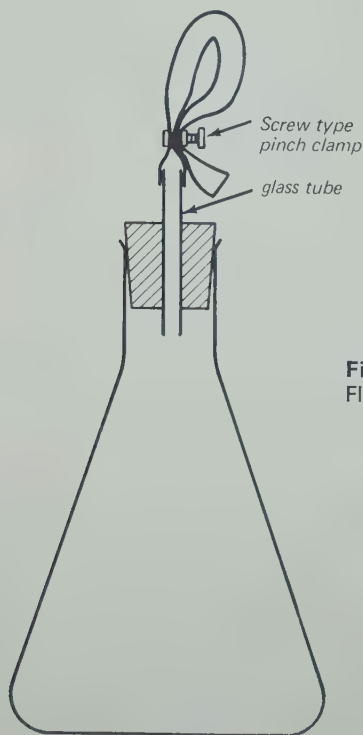


Figure 4

Flask for demonstrating that air has mass.

CLASSROOM PRESENTATION

- Determine the mass of the flask full of air.
- Evacuate the flask and determine its mass. Wear safety glasses in case of an implosion.

- c) Develop the need for determining the volume of air removed from the flask by asking the class if they think all the air has been removed.

If they agree (correctly) that it has not, ask how can the volume removed be determined? Doing this helps them with their volume determination in Experiment 5.

Loosen the pinch clamp under water and let the atmosphere push water into the flask. Adjust water levels inside and outside the flask. Pinch the hose closed, remove the flask from the water, and measure the volume of water. This is equal to the volume of air originally removed.

- d) Determine the mass of one liter of air at the conditions prevailing in your lab. This value can be used by the students when they correct for buoyancy of air in their calculations for Experiment 5. The mass of air removed equals the difference in the masses before and after evacuation.

$$\text{Density of air} = \frac{\text{mass}}{\text{volume}}$$

Students sometimes find the kinetic motion of molecules hard to picture. Demonstration 3 may help, p. 25.

Experiment 5

The Masses of Equal Volumes of Gases

PURPOSE AND SUMMARY

To give experimental evidence that equal volumes of gases do not have equal masses (at the same temperature and pressure) and later, after a discussion of Avogadro's Hypothesis to enable the student to calculate a molar mass from laboratory measurements. The student is thereby able to understand that molar masses are based on experiments which he can perform.

The student weighs a plastic bag assembly empty and also when filled with oxygen, carbon dioxide, and if time allows, another gas. He determines the volume of the full bag of gas by displacing the gas into a jug full of water, and measuring the water required to refill the jug.

TIMING

This experiment should precede any reading assignment in Chapter 2.

About 40 minutes are needed in the lab.

MATERIALS NEEDED**per student or pair**

- 1 quart-size plastic bag (frozen food type)
- 1 one-hole rubber stopper, #5 or #6. See Lab Hint 1.
- 1 rubber band
- 1 medicine dropper
- 1 pneumatic trough or other water container
- 1 half-gallon jar (or 2½ liter acid bottle) plus stopper or glass plate
- 1 rubber delivery tube, 50–60 cm long
- 1 thermometer, –20°C to 150°C
- 1 large graduated cylinder. See Lab Hint 2.

per class

- balances, centigram
- source of O₂, CO₂, and possibly another gas. See Lab Hint 3.

LAB HINTS

1. The groove around the stopper should be shallow and smooth. This can be made by clamping the stopper between two nuts and washers on a threaded rod. Chuck this up in a drill press. While the assembly is rotating, hold a round file against the side. Cut a groove about 2 mm deep and midway up the side of the stopper. If a drill press is not available, a sharp knife lubricated with water is suitable.

Drying of bags between periods can be facilitated if they are separated from the rubber stopper and spread out on paper towels.

2. The directions call for a large graduated container. If there is a shortage of large graduated cylinders, have some calibrated containers such as liter flasks or quart jars available. Mark such containers with their volume and the uncertainty of the calibration. The level of water inside the large collecting bottle should be close to that in the trough, but need not be exactly the same. A 10 cm difference of level makes only a 1% change in pressure.
3. a) OXYGEN

When a supply of compressed oxygen is not available in the lab, the most convenient source for most schools is to borrow a tank from the auto shop or maintenance department. You can transport some gas in a large inner tube. Remove the valve core, attach a rubber hose, fill and close with a pinch clamp.

The small “lecture” bottles hold so little that they are not really very satisfactory. However, two cubic feet—the volume in the usual lecture bottle carried by most supply companies—will be enough for one class (2 cubic feet of O₂ is about 56 liters and costs about \$1.50 if you already own the cylinder and order directly from the manufacturer). Anchor the cylinders securely.

If bottled oxygen is not available, it can be generated by using stabilized 30% H₂O₂. (**CAUTION:** 30% H₂O₂ causes burns. Use safety glasses and gloves. In case of a spill, wash it off with plenty of water.) Set up a generator by inserting a dropping funnel into a two-hole rubber stopper equipped with a delivery tube. The stopper assembly is placed in a 250 ml flask containing a gram or two of MnO₂ powder. Using distilled water, dilute 50 ml of 30% H₂O₂ to 100 ml. Add the diluted H₂O₂ via the dropping funnel as needed to release a steady flow of oxygen. Use a drying tube containing anhydrous CaCl₂ or CaSO₄.

This amount of H_2O_2 will give about $5\frac{1}{2}$ liters of oxygen at room temperature and pressure. For student use, supply H_2O_2 which has already been diluted. A 5% solution reacts rapidly enough and is safer to use. A $\text{KClO}_3\text{--MnO}_2$ mixture is *not recommended*. Many serious accidents have occurred with this combination. Certainly it should not be used by students. If you decide to use it, be sure that both the MnO_2 and the KClO_3 have been heated strongly in evaporating dishes (separately) and that each is known to be free of any organic or other reducing material. Be sure that none of the mixture gets on the rubber stopper of the generator.

b) CARBON DIOXIDE

An inexpensive way to obtain CO_2 gas is from Dry Ice. One hundred students will require 2–4 lb. Place chunks of it in a large flask equipped with a two-hole rubber stopper. In one hole put a 6 cm piece of fire-polished glass tubing, in the other, a glass elbow (6 and 10 cm arms). To operate, direct a heat lamp on the flask. Attach bag to the long arm of the elbow with a short rubber hose. Hold finger over the fire-polished end of the straight tube. Carbon dioxide is forced into the bag. Releasing the finger vents the gas safely to the room. If Dry Ice is not readily available, set up a CO_2 generator using CaCO_3 and HCl . Use a 250 ml flask with a long-stem funnel and a delivery tube with drying tube attached. Add about 25 grams of CaCO_3 and about 100 ml of 6 *M* HCl . This will produce about 6 liters of CO_2 at 25°C and 1 atmosphere. Carbon dioxide is also available in lecture bottles. These contain 4 cubic feet of the gas, which costs \$1.50 excluding cost of the cylinder. While in use, clamp the cylinder in an upright position.

c) OTHER GASES

Since it is desirable to have a gas less dense than air, ammonia is a good choice, but adequate ventilation is essential. Lecture cylinders holding 8 cubic feet cost about \$1.50 excluding the cost of the cylinder. Clamp these vertically also. Another way to obtain ammonia is by warming a dry mixture of NH_4Cl and Ca(OH)_2 . About 100 grams of each will produce enough NH_3 for one class (about 50 liters).

Methane may also be purchased in cylinders. Because of its general availability, natural gas is useful. Finding a bag full of this gas weighs less than the empty bag causes much student interest. In addition to this, problem 2 under *Additional Investigations* is a real challenge to the best students.

PRELAB DISCUSSION

On the day before this experiment is to be done, have a plastic bag fastened to a stopper as described in the directions. Press out all the air, weigh the bag, and fill it with air. To avoid excess moisture, use a hand pump if compressed air is not convenient. Equalize the pressure, put the cap in place, and weigh the bag full of air. When the students have observed the identity ($\pm$ the uncertainty of the measurement) of these two masses, ask: "Would this be true regardless of the gas used to fill the bag?" Do not discuss the answer, but assign the reading of the experiment. If this seems too little for one night, give some questions for review of Chapter 1. The answer to the problem posed in the experiment comes early in the chapter; for this reason, delay assigning reading in Chapter 2.

On the day of the experiment, demonstrate air has mass, and determine the mass of one liter of air. See Demonstration 2. This value can then be used by the student in his calculations.

Urge the better students to use other gases in addition to oxygen and carbon dioxide. Avoid using the symbols O_2 and CO_2 .

Have some students start with oxygen, others with carbon dioxide. This relieves congestion around the generators or tanks.

Remind students to determine the volume of the bag only after all gases they are going to use have been weighed. Otherwise a wet bag must be dried.

Avoid a long presentation about buoyancy. The following may be adequate. A block of wood falling through air is stopped when it hits the water in a swimming pool. Why? Must not the water provide an upward force to counterbalance the force supplied by gravity? The wood sinks until the upward force equals the downward force. In a similar way, a balance gives the net effect resulting from all the forces acting on an object being weighed. The two most important are the attraction of the earth (gravity), pulling the object down, and the upward push due to the displaced material.

Air, like water, is a fluid which imposes an upward force on objects immersed in it. A plastic bag full of oxygen gas experiences an upward force when it is weighed in air. The mass of the oxygen, as determined in air, is then a mass which has been reduced by this upward force. To determine the amount of material contained in the bag, its mass in air will have to be increased to allow for the upward force supplied by the air. This is also true when a person is weighed in air, but the upward force or buoyancy is so much less than the gravitational force that the buoyancy can be ignored.

PRECAUTIONS

Observe the usual lab precautions plus special care if noxious gases are used.

POSTLAB DISCUSSION

The student should see the range of values reported for each ratio asked for in Question 5. This can be done by having the students record their ratios on a wall chart the day after the experiment. Or ratios can be compiled when the write-ups are corrected.

The extent to which uncertainty is treated will vary with the background of the students. The minimum treatment must at least convince the student that mathematics cannot improve the precision of a measurement. The value for the ratio $2.23/1.60$ is 1.39. The answer 1.39375 is scientifically wrong. Although the arithmetic is correct, it contains much more information than the data from which it was obtained.

SAMPLE DATA (At 20°C, 1 atm)

Mass of bag assembly	22.53 ± 0.01 g
Mass of bag assembly + O ₂	22.68 ± 0.01 g
Mass of bag assembly + CO ₂	23.31 ± 0.01 g
Mass of bag assembly + NH ₃	21.93 ± 0.01 g
Volume of bag (1.200 ± 0.025) liters.	

The precision of the volume measurement will be quite variable depending, in part, on the size of the volumetric equipment available. Assuming 12 measurements are made with a 100 ml graduated cylinder, it could be ± 6 ml. But since the level in the large container cannot be marked exactly, a ± 25 ml estimate has been used.

RESULTS OF PROCESSING DATA

- 1) What is the apparent mass of oxygen in the bag? (Subtract the mass of the empty bag from the mass of the bag full of oxygen.)

From the sample data, we have

$$\begin{array}{r} 22.68 \pm 0.01 \text{ g} \\ - 22.53 \pm 0.01 \text{ g} \\ \hline 0.15 \pm 0.02 \text{ g} \end{array}$$

- 2) Calculate the mass of the air displaced by the bag filled with gas.

From the data above and from the table, we find

$$(1.20 \text{ liters} \pm 2\%) \times (1.21 \text{ g} \pm 1\%)/\text{liter} = 1.45 \text{ g} \pm 2\%$$

Since 2% of 1.45 g is 0.03 g, we have $1.45 \pm 0.03 \text{ g}$.

- 3) What is the actual mass of oxygen in the bag? (Add the mass of the air displaced to the apparent mass recorded from the balance.)

$$(1.45 \pm 0.04 \text{ g}) + (0.15 \pm 0.02 \text{ g}) = 1.60 \pm 0.06 \text{ g}$$

or

$$1.60 \text{ g} \pm 4\%$$

- 4) In a similar way determine the actual mass of carbon dioxide gas in the bag and the actual mass of any other gas used.

For carbon dioxide:

Apparent mass

$$\begin{array}{r} 23.31 \pm 0.01 \text{ g} \\ - 22.53 \pm 0.01 \text{ g} \\ \hline 0.78 \pm 0.02 \text{ g} \end{array}$$

Actual mass of CO_2

$$(1.45 \pm 0.04 \text{ g}) + (0.78 \pm 0.02 \text{ g}) = 2.23 \pm 0.06 \text{ g, or } 2.23 \text{ g} \pm 3\%$$

For ammonia:

Apparent mass

$$\begin{array}{r} 21.93 \pm 0.01 \text{ g} \\ - 22.53 \pm 0.01 \text{ g} \\ \hline -0.60 \pm 0.02 \text{ g} \end{array}$$

Actual mass of ammonia

$$(1.45 \pm 0.04 \text{ g}) + (-0.60 \pm 0.02 \text{ g}) = 0.85 \pm 0.06 \text{ g, or } 0.85 \text{ g} \pm 7\%$$

- 5) Compare the actual mass of each gas measured to that of oxygen by dividing each mass by the mass of the same volume of oxygen. Express each ratio as a decimal fraction.

$$\text{Mass of } \text{CO}_2 \text{ to } \text{O}_2 \quad \frac{2.23 \text{ g} \pm 2\%}{1.60 \text{ g} \pm 4\%} = 1.39 \pm 4\% \text{ or } 1.39 \pm 0.06 \text{ g}$$

$$\text{Mass of } \text{NH}_3 \text{ to } \text{O}_2 \quad \frac{0.85 \text{ g} \pm 7\%}{1.60 \text{ g} \pm 4\%} = 0.53 \pm 7\% \text{ or } 0.53 \pm 0.04 \text{ g}$$

A QUESTION TO WONDER ABOUT

Is there any relation between the masses of equal volumes of gases and the relative mass of molecules?

After Section 2-3 has been assigned in the Textbook, remind the students of this question. Exercise 2-2 is based directly on this experiment.

ADDITIONAL INVESTIGATIONS

—to be undertaken as extracurricular experiments.

- 1) How do the masses of equal volumes of gases at atmospheric pressure change as the temperature is increased?

Let the exceptional student who decides to tackle this problem suggest something first. One possibility is to drill a hole in the base of a balance (± 0.01 g) that may be placed on top of a drying oven. Remove the pan and pan support and, after proper counterbalancing, suspend a plastic bag from the hook by a thread extending through the hole in the support and into the oven. The bag may be filled with oxygen and later with CO_2 or other gases and fitted with a cap such as the one used in Experiment 5, except that a hole should be made in it with a hot needle in order that the pressure may be equalized as the gas is warmed. A graph of the changes in mass during the warming of each gas should make an interesting record. When a constant mass is reached at a given temperature, you may assume that the gas is at the same temperature and pressure as the oven.

- 2) The burner gas in the laboratory is usually natural gas, which is mostly methane, CH_4 , but contains some ethane, C_2H_6 , and small amounts of other compounds. Weigh a sample of burner gas and calculate the mole percent of methane and of ethane, assuming that they are the only gases present.

It is assumed that the student will not get to this immediately after the completion of the experiment and that he will have read all of Sections 2-7 and 2-8 by the time he considers this problem. The mass ratio of natural gas to oxygen is usually about 0.58 to 1, which gives a "molar mass" of about 19 and a molar composition of 80% methane and 20% ethane.

Let x = mole % of methane. Then $100 - x$ = mole % ethane.

$$\frac{16x}{100} + \frac{30(100 - x)}{100} = 19$$

$$\begin{aligned} 16x + 3000 - 30x &= 1900 \\ -14x &= -1100 \end{aligned}$$

$$x = \frac{1100}{14} = 78 \text{ mole \% of methane}$$

DEMONSTRATION 3**KINETIC MOTION**

You may use Ping-Pong balls in a screened cage to illustrate the motions of gas particles. The apparatus could be a bird cage containing a wire mesh floor. When air is blown in the bottom by an electric fan, the motion of the balls as they strike the walls of the cage will be analogous to the invisible air particles pushing against the walls of the balloon.

A quick demonstration of the kinetic model can be made with a book and a pencil. Suggest that the book is part of the container wall and the eraser end of the pencil is a molecule. Then tip the book slightly, and tap it lightly but rapidly. It will be held up by the colliding molecules. More frequent or stronger taps will hold the book more upright, illustrating increased pressure. A fairly heavy book works best. If the book is too light, you will find it troublesome trying to avoid knocking it over.

Practice a few times before you use this demonstration. In fact, never present any demonstration without practice.

DEMONSTRATION 4**PARTICLE MODEL OF GASES**

Another approach to use in explaining the gaseous phase consists of applying the model to an inner tube. All students know that tires require air for support. Take a deflated tube, and show how easy it is to press the walls together. You say, "It contains no air? Let us see." Remove the valve core, attach a piece of rubber tubing, and press on the inner tube while the end of the tubing is held under water.

The air left in the tube is at the same pressure as that outside. A "flat tire" is full of air, but the air is not at a high enough pressure to support a car or bicycle.

Insert the valve core, and pump air into the tube. Try pressing the tube against the table. You will notice that something inside the tube resists compression. Referring to your previous models, ask the students if they can see a relationship between the forces demonstrated in the models and the resisting force in the tube. Many relationships of gases can be used here, such as pressure varying with the number of particles, the number of collisions between particles, or the energy of motion for particles.

DEMONSTRATION 5**SOLUBILITY OF AMMONIA GAS OR HYDROGEN CHLORIDE GAS**

A convenient way to demonstrate the solubility of these gases is to use 50 ml disposable plastic syringes available through medical supply houses. Fill a syringe with gas by attaching the syringe to a lecture bottle using a short piece of Tygon tubing. By carefully controlling the valve on the gas bottle you can collect a syringe-full of the gas without blowing the plunger and letting gas into the classroom.

Place the outlet of the gas-filled syringe into a beaker of water. Pull out on the plunger to bring water in contact with the gas. The reaction is quite sudden. The plunger is forcibly pulled from your grasp and is slammed downward into the syringe.

DEMONSTRATION 6**REACTION OF AMMONIA AND HYDROGEN CHLORIDE**

Fill two syringes, one with ammonia gas and the other with hydrogen chloride gas. Join the two syringes with a short piece of Tygon tubing. Push one gas into the other. An immediate reaction is visible. You can do quantitative experiments using various amounts of the gases to determine the ratio of their combining volumes. Be sure the syringes are dry before you fill them with the gases.

DEMONSTRATION 7**CONSTRUCTION OF MOLECULAR MODELS**

Section 2-6 deals with models. Several kinds are presented to make the point that a given kind shows only certain features and fails to show others. Chemists use different kinds for different purposes. An important point to make is that any model is just what the name implies. It gives only a partial representation, and this is usually flawed. For example, molecules do not have definite boundaries, as do our models.

The following directions will permit you to make models of a dozen or so substances frequently referred to in the Textbook. The plan is essentially this: Styrofoam balls of the proper diameter (van der Waals) are cut so as to give proper separation (fixed by covalent radius) when glued together.

Before giving the details, let us describe the source of data. For the distance between bonded atoms we use covalent radii. These are deduced from interatomic distances in molecules, as

determined by the methods of X-ray diffraction, electron diffraction, and molecular spectroscopy. Pauling* (and others) have attempted to divide these bond lengths into additive contributions from each atom. The additivity of such assigned radii is not perfect, but it is sufficiently good so that the radii have wide acceptance among chemists. Note that we *never* measure a radius; we always measure an internuclear distance. Furthermore, X-ray measurements of solids are not as good as those made on gaseous molecules, hence they are used *only* when reliable gas phase work is not available—which is often, of course. For the sphere diameter we use the van der Waals radii determined from interatomic distances between nonbonded atoms in crystals. The result is a “distance of nearest approach” for nonbonded atoms. These are also given by Pauling. The table gives some values. For actual construction, some scale must be chosen. A scale of $2\text{ cm} = 1\text{ \AA}$ gives a size that is convenient for classroom use. This makes the model 200 million times as large as the actual molecule.

Some Covalent and van der Waals Radii

Element	Covalent Radii (r_c in $\text{\AA}$)	van der Waals Radii (r_v in $\text{\AA}$)	Angle Between Bonds (degrees)
hydrogen	0.30	1.1	—
hydrogen (in H_2)	0.37	1.1	—
carbon (single-bonded)	0.77	1.7	109.5
nitrogen (single-bonded)	0.70	1.50	109
nitrogen (in N_2)	0.55	1.50	—
oxygen (in O_2)	0.60	1.40	—
oxygen (two single bonds)	0.66	1.40	105
sulfur (two single bonds)	1.04	1.85	92
fluorine	0.64	1.35	—
chlorine	0.99	1.80	—

The construction of atom models is divided according to the number of “bonds”—that is, according to how many segments are cut off. The procedure is the same for all “one-bond” atomic models (H, Cl, F), although different dimensions must be used. Likewise for “two-bond” (O, S), three-bond (N), and four-bond (C) models.

PROCEDURE: GENERAL

Select the Styrofoam sphere. If the proper diameter is not available, an oversized one can be rolled down. The reduction in diameter is limited to a few millimeters, depending on the sphere diameter and the density of the Styrofoam. Use books or piles of paper to get the proper height, then roll with a piece of plywood resting on the books. This produces a compact and more durable surface. In addition, it is easy to paint and looks better.

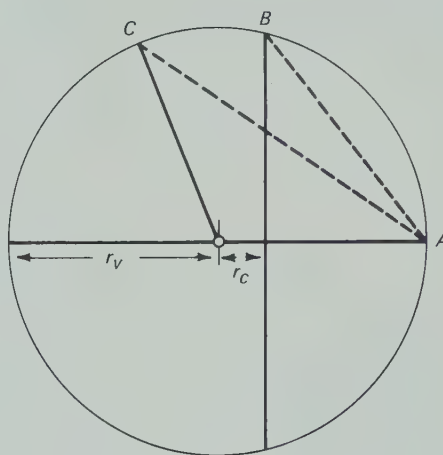
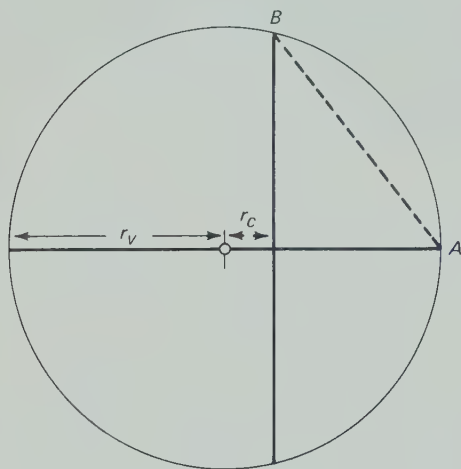
An easy way to establish the position of the cut is to construct plane figures as follows.

* L. Pauling, *The Nature of the Chemical Bond*, Cornell Univ. Press (1960), Chapter 7. Useful tables are on pp. 224, 256, and 260.

Model of Atom with a Single Bond

- Draw a circle with radius equal to the van der Waals radius. Use whatever scale you choose; all values in the table on page 27 will be doubled if you use $2\text{ cm} = 1\text{ \AA}$. Mark the center.
- Draw a diameter of the circle.
- Measure out one r_c from the center, and draw a perpendicular.

The results should be as shown in the diagram below at the left.



- Now set a compass for the distance AB , and draw a circle on the sphere using this radius.
- Use a hacksaw blade to saw the segment off, leaving a few millimeters extra on the sphere. Sand this off with fairly coarse garnet paper. You can use sandpaper, but it is less satisfactory. Proceed gently; the Styrofoam cuts very easily. *Note:* Painting can be done before or after assembly.
- Paint the curved surfaces with white latex-base paint. This acts as filler and undercoat to protect the plastic, which dissolves in some enamels.
- Enamel is best for the second coat of paint.

Model of Atom with Two Bonds

Repeat steps a–c as for the single-bonded atomic model.

- Construct a radius from the center at the angle given in the table on page 27. The results are as shown in the diagram above at the right.
- Mark a dot on the sphere.
- Set AC on the compass, and lay it off from the dot. Make a second dot on the sphere.
- Now set AB on the compass, and draw a circle around each dot.
- Saw, sand, and paint as before. Be careful that sanding maintains the angle.

Models of Atoms with Three or Four Bonds

Repeat steps a–f as for double-bonded atomic models.

- g) Using the two dots as centers and AC as the compass setting, locate one (or two) more dots, equidistant from the two marked. Draw short arcs from each marked spot to get the new point at the intersection(s).
- h) Now set AB on the compass, and draw a circle around each point on the sphere.
- i) Saw, sand, and paint as before.

PROCEDURE: ASSEMBLY

After the models are prepared, there are two ways to assemble them. For “permanent” models, glue the flat faces together with a few drops of white plastic glue (Elmer’s or Wilhold are good). A few models, particularly two of H_2 and one of O_2 , should come apart easily, for illustrating the formation of water and the balancing of chemical equations (Chapter 3). For this, two modifications are needed. Some kind of peg is required for holding the atomic models together, and half the hydrogens must be hollowed out to fit on a spherical part of an oxygen. For the peg, use a piece of chenille stem glued in at one end. To hollow out an atom model, first cut the flat face, gouge some out with a knife or your fingernails. Then, finish it off by holding a $\frac{1}{2}$ inch strip of garnet paper over the oxygen-size sphere and gently rotating the hydrogen. Some extra chenille stem “bonds” are required. With a compass point, punch a hole in the oxygen for the “bond” to a hydrogen.

REFERENCE INFORMATION

Suppliers

Of the many suppliers of Styrofoam, those below are given as sources of three typical types of spheres. Others may be found listed in the yellow pages of a phone book under Plastics, Foam. Send for price lists.

Raytheon Education Company, 186 Third Avenue, Waltham, Mass. 02154 (molecular model kit).

Star Band Co., Broad and Commerce Sts., Portsmouth, Va. (abraded spheres which can be compressed to obtain great range of diameters).

Foam Plastics Inc., Forest Lake, Minn. (molded spheres, compressible, smooth surface).

Plasteel Corp., 26970 Princeton, Inkster, Mich. (molded pellets, noncompressible).

Data for other models

L. Pauling, *The Nature of the Chemical Bond*, Cornell Univ. Press (1960).

A. F. Wells, *Structural Inorganic Chemistry*, Clarendon Press (Oxford Univ. Press) (1962).

Tables of Interatomic Distances and Configuration in Molecules and Ions, Special Publ. #11. See also the *Supplement*, Special Publ. #18 (1965). The Chemical Society, Burlington House, London, 1958.

Uses

Numerous articles in *J. Chem. Ed.* have described uses and ways to make models.

DEMONSTRATION 8**THE SIZE OF A MOLE OF WATER
AS A LIQUID AND AS A GAS**

PURPOSE

To provide a scale model of the molar volume of a common substance, water.

TIMING

During Section 2-7 and throughout the course.

MATERIALS NEEDED**for liquid molar volume, 18 cm^3**

1 cube of wood 2.6 cm on each edge, painted blue. For 0°C , the cube edge length is 2.6216 cm and for 25°C , 2.6241 cm.
or one sheet heavy paper or light cardboard $4'' \times 5''$.

for gas molar volume, 22.4 liters

1 cube of wood, 28.2 cm each edge painted white or one sheet poster board, $3' \times 4'$.

PREPARATION OF MODELS

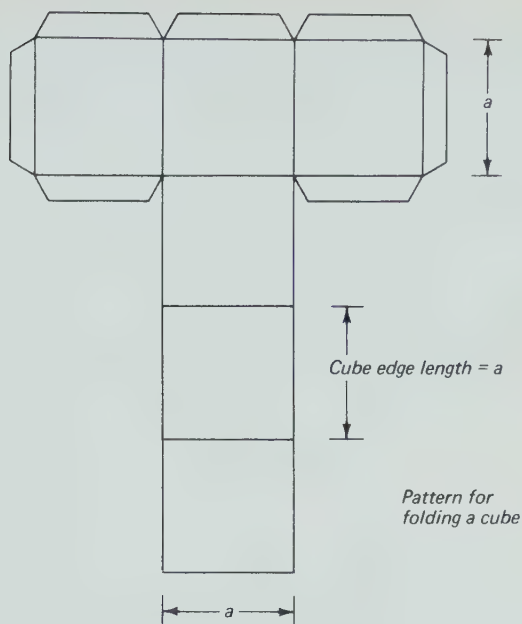
The small wooden model can be cut from any piece of wood thicker than about $1\frac{1}{4}$ inches. For the 22.4 liter model, cut six square pieces of plywood 11.10 inches on the side. Bevel the edges at 45° and glue the sides into a cube. Fill cracks and paint.

The cardboard versions are less durable but easier to make, especially for the larger cube. Draw a full scale pattern as shown in the diagram on the next page, cut out the shape, and score the internal lines. Fold and glue.

You may find the shop or art teacher helpful in these projects.

CLASSROOM PRESENTATION

Use the small model to illustrate the volume of a mole of liquid water. Later, in Chapter 4, when molar volume of gases is discussed, these cubes are very useful for contrasting the volume available to a molecule in the liquid phase, with the volume in the gas phase.



Experiment 6

Iron-Copper Sulfate Reaction

PURPOSE AND SUMMARY

To determine a quantitative relation between moles of iron used and copper formed when iron filings react with copper sulfate solution. To emphasize the mole concept and provide a basis for equation writing later on.

The student adds iron filings to an excess of copper sulfate in solution. He determines the mass of iron used and the mass of copper produced. From these data he is able to determine the mole relation for the reaction.

TIMING

After Section 2-9 in the Textbook.

About 40 minutes are required to make the weighings. Let the reaction reach equilibrium, and wash the copper making it ready to be dried. This convenient stopping point occurs after step h. A few minutes are required during a subsequent period to weigh the dried copper.

MATERIALS NEEDED**per student or pair**

- 1 beaker, 100 ml
- 10 g copper sulfate pentahydrate, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$
- 2 g iron filings, fine mesh
- 1 piece of weighing paper
- 1 stirring rod, glass
- 1 wash bottle
- 1 ring stand, iron ring, and wire gauze
- 1 burner

per class

- balances, centigram
- drying oven

LAB HINTS

1. Drying can be conveniently carried out in a drying oven a little above 100°C . Do not overheat the copper since it will oxidize at higher temperatures. If other methods of drying are used, be careful not to overheat the samples.
2. It is convenient to have reagent bottles of copper sulfate and iron filings at each balance station.
3. Pieces of "slick" magazine pages make excellent weighing papers. Do not use filter paper or paper towels.

PRELAB DISCUSSION

Little discussion is needed. Some instruction is probably needed on weighing approximate amounts and on the use of weighing paper.

PRECAUTIONS

Caution the students not to overheat the copper sulfate solution causing it to boil over. Add iron filings cautiously to avoid excessive boiling.

POSTLAB DISCUSSION

The students should encounter little difficulty in making the calculations. However, if some do have difficulty, it is better to let them struggle with the problems on their own before giving them too much assistance.

It is interesting and informative to have the students record both the masses of iron and copper and the ratio of moles of Fe to moles of Cu on a class chart. This shows clearly how different experimental numbers (masses) can yield closely similar derived numbers (the ratios).

This is a good time to stress again the importance of using the correct number of significant figures in all the measurements and calculations.

You may want to collect class data (*Processing the Data* numbers 2 and 3) and make a graph with moles of Fe on one axis and moles of Cu on the other. The resulting scattergram should

clearly indicate the one to one mole relation. It should also indicate the uncertainty. This is an excellent opportunity to discuss the increase in *certainty* obtained when conclusions are based on many determinations rather than on a single one. It also shows that the reaction is reproducible within well-defined limits of uncertainty.

SAMPLE DATA

Mass of empty beaker	$37.55 \pm 0.01 \text{ g}$
Mass of weighing paper	$1.03 \pm 0.01 \text{ g}$
Mass of iron filings plus weighing paper	$3.05 \pm 0.01 \text{ g}$
Mass of product (dry solid) plus beaker	$39.90 \pm 0.01 \text{ g}$

RESULTS OF PROCESSING THE DATA

- 1) Determine the mass of the product.

$$\begin{array}{r}
 \text{mass of product plus beaker} \quad 39.90 \pm 0.01 \text{ g} \\
 - \text{mass of beaker} \quad 37.55 \pm 0.01 \text{ g} \\
 \hline
 \text{mass of product} \quad 2.35 \pm 0.02 \text{ g}
 \end{array}$$

- 2) Determine the moles of iron used. Recall that the mass of a substance divided by the mass per mole equals the number of moles.

$$\text{mass of iron used} = (3.05 \pm 0.01) - (1.03 \pm 0.01) = 2.02 \pm 0.02 \text{ g.}$$

$$\text{moles of iron used} = \frac{2.02 \pm 1\% \text{ g}}{55.8 \text{ g/mole}} = 0.0362 \pm 1\% \text{ mole}$$

- 3) Assuming the product to be copper, determine the moles of copper produced.

$$\text{moles of copper produced} = \frac{2.35 \pm 1\% \text{ g}}{63.5 \text{ g/mole}} = 0.0370 \pm 1\% \text{ mole}$$

- 4) Divide the moles of iron, Fe, by the moles of copper, Cu, to determine the mole ratio, Fe/Cu. Be sure to express your answer as a decimal and use the correct number of significant figures.

$$\text{moles of Fe/moles of Cu} = \frac{0.0362 \pm 1\%}{0.0370 \pm 1\%} = 0.98 \pm 1\%$$

- 5) What evidence did you observe to indicate that some of the copper in solution remained unchanged?

The solution retained some blue color after all the iron is used up.

ADDITIONAL INVESTIGATIONS

Heat the copper obtained in the experiment to form the oxide of copper. Heat the copper strongly several times and stir the solid carefully so that it can react with oxygen in the air. Allow it to cool and weigh it to determine the mass of the oxide. Heat and cool it again until a constant mass is obtained. Determine the mass of oxygen that has combined with the copper. Determine the mole ratio, Cu/O, for the compound formed.

Experiment 7

Copper-Silver Nitrate Reaction

PURPOSE AND SUMMARY

To provide an experimental determination of the molar relation shown when silver and copper react. To emphasize the mole concept and provide a basis for equation writing.

The student allows copper wire to react with silver nitrate solution to obtain silver crystals and a solution of cupric nitrate. He determines the change in mass of the copper wire, and the mass of the silver produced. From these data he is able to determine the mole relation for the reaction.

TIMING

After Section 2-9 in the Textbook.

Overall time is about one full laboratory period plus 10 or 15 minutes from two other periods; one before and one after the main session.

The reaction between the copper wire and the silver nitrate solution takes a full 30 minutes. A short period, 10 to 15 minutes, the day before the experiment will allow the students to weigh the wire, the beaker, and the vial of silver nitrate. They can also coil the copper wire as directed in step d to be all ready to begin the next day with step c.

On the day of the experiment it should take about 10 minutes to get set up and about 30 minutes for the reaction. An additional 5 to 10 minutes are needed to wash the silver crystals and prepare them for drying.

An additional 10 to 15 minutes are needed on a subsequent day to weigh the dried silver crystals and start Experiment 8.

MATERIALS NEEDED

per student or pair

- 30 cm length of bare copper wire, #16 or #18 gauge
- 1 beaker, 100 ml
- 1 beaker, 250 ml (for waste solution)
- 1 test tube, 18 × 150 mm
- 1 vial of silver nitrate fine crystals, AgNO_3 , about 3 grams
- 1 stirring rod, glass
- 1 stopper, rubber, one-hole, #3
- 1 graduated cylinder, 25 ml
- 1 ring stand and clamp
- 1 watch glass, cover for 100 ml beaker
- 1 wash bottle

per class

balances, centigram

several acetone baths

drying oven

about 1 *M* Na₂S₂O₃ solution to remove Ag⁺ and prevent stains.

LAB HINTS

Drying can be accomplished conveniently and quickly using a drying oven. An alternate method is to use two or three infrared heat lamps mounted in the fume hood.

PRELAB DISCUSSION

Little discussion is needed especially if the students have done Experiment 6. If they did not do Experiment 6, some instruction is probably needed on weighing.

If the students are still having weighing problems, an easy way of checking is to have them report the mass of the 30 cm length of copper wire. The diameter of the wire is constant enough that if the length is measured and cut carefully you can be quite certain of the mass of each piece.

PRECAUTIONS

Warn the students about getting silver nitrate, solid or solution, on their hands and clothing. If it is spilled on either hands or clothing, the immediate use of sodium thiosulfate (hypo) solution may prevent staining. Wash the hypo off the clothing with water.

POSTLAB DISCUSSION

The students should not encounter any difficulty in making the calculations, especially if they have done Experiment 6. Let them try all the calculations on their own before offering assistance.

It is interesting to have the students record the masses of Ag and Cu and the ratio $\frac{\text{moles Ag}}{\text{moles Cu}}$ on a class chart. This shows clearly how different experimental numbers (masses) can yield closely similar derived numbers (the molar ratio, Ag/Cu).

This is a good time to stress again the importance of using the correct number of significant figures in all the measurements and calculations.

You may want to collect class data and make a graph showing moles of Ag on one axis and moles of Cu on the other. The resulting scattergram should show the two-to-one relation. It should also indicate the uncertainty. This is an excellent opportunity to discuss the increase in *certainty* obtained when conclusions are based on many determinations rather than on a single one. It also shows that the reaction is reproducible within well-defined limits.

SAMPLE DATA

Mass of clean dry 100 ml beaker	36.93 ± 0.01 g
Mass of vial, cap, and silver nitrate	15.36 ± 0.01 g
Mass of empty vial with cap	12.35 ± 0.01 g
Mass of the coiled copper wire before reaction	2.72 ± 0.01 g
Mass of the coiled copper wire after reaction	2.16 ± 0.01 g
Mass of the beaker plus silver crystals	38.83 ± 0.01 g

RESULTS OF PROCESSING THE DATA

- 1) Determine the change in mass of the copper wire during the experiment.

$$\begin{aligned}\text{Change in mass} &= (2.72 \pm 0.01 \text{ g}) - (2.16 \pm 0.01 \text{ g}) \\ &= 0.56 \pm 0.02 \text{ g} = 0.56 \text{ g} \pm 4\%\end{aligned}$$

- 2) Determine the number of moles of copper which reacted. Recall that the mass of a substance divided by the mass per mole equals the number of moles.

$$\begin{aligned}\text{moles of copper reacted} &= \frac{0.56 \text{ g} \pm 3.6\%}{63.5 \text{ g/mole}} = 0.0088 \text{ mole} \pm 4\% \\ &= 8.8 \times 10^{-3} \text{ mole} \pm 4\%\end{aligned}$$

- 3) Determine the mass of silver obtained.

$$\begin{aligned}\text{mass of silver} &= (38.83 \pm 0.01 \text{ g}) - (36.93 \pm 0.01 \text{ g}) \\ &= 1.90 \pm 0.02 \text{ g} \\ &= 1.90 \text{ g} \pm 1\%\end{aligned}$$

- 4) Determine the number of moles of silver.

$$\begin{aligned}\text{moles of silver} &= \frac{1.90 \text{ g} \pm 1\%}{108 \text{ g/mole}} = 0.0176 \text{ mole} \pm 1\% \\ &= 1.76 \times 10^{-2} \text{ mole} \pm 1\%\end{aligned}$$

- 5) Divide the moles of silver by the moles of copper to determine the ratio Ag/Cu. Be sure to express your answer as a decimal and to the correct number of significant figures.

$$\text{moles Ag/moles Cu} = \frac{1.76 \times 10^{-2} \pm 1\%}{8.8 \times 10^{-3} \pm 4\%} = 2.0 \pm 4\%$$

- 6) Determine the moles of AgNO₃ used in the experiment.

$$\begin{aligned}\text{moles AgNO}_3 \text{ used} &= \frac{\text{mass AgNO}_3 \text{ used}}{170 \text{ g/mole}} \\ &= \frac{3.01 \text{ g} \pm 0.7\%}{170 \text{ g/mole}} = 1.77 \times 10^{-2} \text{ mole} \pm 0.7\%\end{aligned}$$

- 7) Divide the moles of silver by moles of silver nitrate to determine the ratio Ag/AgNO₃. Express your answer as a decimal using the correct number of significant figures.

$$\begin{aligned}\text{moles Ag/moles AgNO}_3 &= \frac{1.76 \times 10^{-2} \pm 1\%}{1.77 \times 10^{-2} \pm 0.7\%} \\ &= 0.99 \pm 1\%\end{aligned}$$

- 8) Using the results of your experiment, write whole number coefficients in the following statement:

One mole of Cu plus 2 mole(s) of AgNO₃ in solution produces 2 mole(s) Ag plus one mole of Cu(NO₃)₂ in solution.

- 9) How many atoms of copper were removed from the wire used in your experiment?

$$\begin{aligned}\text{atoms of copper removed} &= \text{moles of copper} \times 6.0 \times 10^{23} \frac{\text{atoms}}{\text{mole}} \\ &= 8.8 \times 10^{-3} \text{ mole} \times 6.0 \times 10^{23} \text{ atoms/mole} \\ &= 5.3 \times 10^{21} \text{ atoms of copper}\end{aligned}$$

- 10) How many atoms of silver metal were formed in your experiment?

$$\begin{aligned}\text{atoms of silver formed} &= \text{moles of Ag} \times 6.0 \times 10^{23} \text{ atoms/mole} \\ &= 1.76 \times 10^{-2} \text{ mole} \times 6.0 \times 10^{23} \text{ atoms/mole} \\ &= 10.6 \times 10^{21} \text{ atoms of silver}\end{aligned}$$

QUESTIONS TO WONDER ABOUT

What caused the color in the solution to appear as the reaction proceeded?

Some of the students will deduce or guess that the color is due to copper in water solution. Have them compare this color to that of a copper chloride solution like that used in Experiment 2 or show a Cu(NO₃)₂ solution.

Experiment 8

Conservation of Mass During Chemical Change

PURPOSE AND SUMMARY

To provide evidence for the law of conservation of mass. To provide additional experience with the mole concept and additional basis for equation writing. To develop some common laboratory techniques.

The silver from Experiment 7 is dissolved in nitric acid and precipitated as silver chloride. The student compares the mass of reactants to mass of products.

TIMING

Start the experiment as soon as Experiment 7 is completed. Have the students add 6 *M* HNO₃ to the silver obtained in Experiment 7. The reaction is slow but can be left overnight in the fume hood to react. Heating to speed the reaction is not recommended except to start the reaction if needed. Too much heating will cause the liquid to evaporate before all of the silver is digested.

The experiment should take about 1½ periods spread over three days.

First Day—10 to 15 minutes, steps a and b.

Second Day—full period, steps c through l.

Third Day—10 to 15 minutes, step m.

MATERIALS NEEDED

per student or pair

2 beakers, 100 ml (one contains silver from Experiment 7)

1 stirring rod, glass

1 funnel and support

1 piece of filter paper. See Lab Hint 5.

1 graduated cylinder, 25 ml

1 wash bottle

1 burner, ring stand, ring, and wire gauze

10 ml 6 *M* nitric acid (384 ml conc. reagent HNO₃/liter)

2 or 3 g sodium chloride, solid

per class

balances, centigram

arrangement for drying. See Lab Hint 1.

LAB HINTS

1. The product in beaker #1 from step b must not be dried in an oven. The fumes will cause severe corrosion. Two or three infrared heat lamps mounted in the fume hood make the most satisfying arrangement. In some climates, overnight drying near an open window may be sufficient.

The silver chloride in beaker #1 from step k and the filtrate in beaker #2 from step l can both be dried in an oven. The filtrate in beaker #2 may be concentrated by heating on a hot plate or with a burner and then placed in an oven for the final drying. The solution tends to spatter if it is dried completely on a hot plate or burner.

2. The silver nitrate produced in Part I may have a little copper nitrate impurity. There may have been some bits of copper present in the silver crystals from Experiment 7. This will not interfere in the experiment since copper will not be precipitated with the silver chloride in Part II. It could, however, be a source of error in the comparison of final product to the silver nitrate obtained in Part I. It will usually be small compared to other errors.

3. Heating the silver chloride precipitate in step h is important in order to coagulate the precipitate for washing. Even though there is a small amount of peptization of the precipitate due to washing with water, it is preferred to washing with dilute nitric acid which causes severe corrosion to the drying oven.
4. The silver chloride from this experiment should be saved and dissolved in sodium thio-sulfate for use in Experiment 36.
5. Have one student weigh a large, known number of pieces of filter paper. Post the mass per piece.

PRELAB DISCUSSION

Students will begin this experiment immediately upon completion of Experiment 7; so little discussion is necessary. You may wish to emphasize the need to learn good laboratory techniques as they are presented, but be sure to avoid discussing expected experimental results.

PRECAUTIONS

See the previous precautions about silver nitrate, Lab Guide p. 35. Caution students concerning the use of 6 M nitric acid. Any spills should be flooded with lots of water and neutralized with sodium hydrogen carbonate solution. Have bottles of about 3 M NaHCO_3 solution (about 250 g per liter) handy. Warn the students also about inhaling the reddish-brown fumes. They are TOXIC. The acid should be added in the fume hood. Wear safety glasses.

POSTLAB DISCUSSION

Collect data from all students for the ratio

$$\frac{\text{mass (AgCl + residue)}}{\text{mass (AgNO}_3 + \text{NaCl)}}$$

so that an average can be determined. You may wish to make a histogram of the ratios so that a central tendency may be clearly recognized.

Discuss the other calculations in *Processing The Data* and the *Questions to Wonder About*.

SAMPLE DATA

Mass of beaker #1 used in Experiment 7	$36.93 \pm 0.01 \text{ g}$
Mass of silver nitrate used in Experiment 7	$3.01 \pm 0.02 \text{ g}$
Mass of silver from Experiment 7	$1.90 \pm 0.02 \text{ g}$
Mass of beaker #1 and solid silver nitrate made in Part I of Experiment 8	$39.96 \pm 0.01 \text{ g}$
Mass of beaker #1, filter paper, and solid silver chloride	$39.44 \pm 0.01 \text{ g}$
Mass of beaker #2	$37.21 \pm 0.01 \text{ g}$
Mass of one filter paper (Lab Hint 5)	$0.58 \pm 0.01 \text{ g}$
Mass of beaker #2 and solid residue	$39.95 \pm 0.01 \text{ g}$

RESULTS OF PROCESSING THE DATA

- 1) Reproduce this table in your notebook and make the necessary calculations to fill in the entries.

	mass (g)	No. of moles
Ag from Experiment 7	1.90	1.76×10^{-2}
AgNO ₃ used in Experiment 7	3.01	1.77×10^{-2}
AgNO ₃ produced in Experiment 8	3.03	1.78×10^{-2}
NaCl	2.19	3.75×10^{-2}
AgCl in beaker #1 (be sure to subtract mass of filter paper) ₁	2.51	1.75×10^{-2}
residue in beaker #2	2.74	no entry

- 2) How does the mass of AgNO₃ produced in this experiment compare with the mass used in Experiment 7? How do you account for any similarity or difference?

The masses should agree quite well unless Ag was lost in Experiment 7. In such a case, the low ratio Ag/Cu for Experiment 7 would give an indication that Ag was lost. Errors in weighing may also be important since the students have had limited experience using the balances.

Students should expect the masses to be the same if no silver was lost. An excess of nitric acid was used in the experiment so that all the silver would form silver nitrate.

- 3) Determine the value of the ratio:

$$\frac{\text{mass (AgCl + residue)}}{\text{mass (AgNO}_3\text{ + NaCl)}}$$

Express your answer as a decimal using the correct number of significant figures. Compare your results with those from others in your class. What can you conclude?

$$\text{ratio} = \frac{5.25 \pm 1\%}{5.22 \pm 1\%} = 1.01 \pm 1\%$$

The comparison should reveal that most students had a ratio near one. Mass was conserved.

- 4) Compare your results for the number of moles of silver used, of silver nitrate produced in Part I, and silver chloride produced in Part II. This comparison can be made by computing the ratio between moles of silver and each of the other substances. Use whole numbers to express your comparison. What can you conclude about the number of moles of silver containing compounds in this series of chemical changes?

$$\frac{\text{moles of Ag}}{\text{moles of AgNO}_3} = \frac{1.76 \times 10^{-2} \pm 1\%}{1.78 \times 10^{-2} \pm 1\%} = 0.99 \pm 1\%$$

$$\frac{\text{moles of Ag}}{\text{moles of AgCl}} = \frac{1.76 \times 10^{-2} \pm 1\%}{1.75 \times 10^{-2} \pm 1\%} = 1.01 \pm 1\%$$

- 5) Calculate the percentage of silver reclaimed in this experiment. Start with moles of AgNO_3 used in Experiment 7 and finish with moles of AgCl produced in Experiment 8.

$$\begin{aligned}\% \text{ Ag reclaimed} &= \frac{\text{moles AgCl obtained in Expt. 8}}{\text{moles Ag used in Expt. 7}} \times 100 \\ &= \frac{1.75 \times 10^{-2} \pm 1\%}{1.76 \times 10^{-2} \pm 1\%} \times 100 = 99 \pm 1\%\end{aligned}$$

QUESTIONS TO WONDER ABOUT

Pure silver nitrate is a white solid. How do you account for any color which may have been present in your sample?

Some students will observe a blue color. They may recall the blue color of copper containing solutions and suggest the presence of some copper compound as an impurity. The silver crystals were either not washed properly or a few flecks of copper were mixed in with the silver.

What causes silver compounds to darken in color when exposed to light?

This question is best left unanswered at this time. It is sufficient to draw attention to the observation and establish the fact that light is responsible for the change. *International Science and Technology*, June 1965, has an excellent article, "The Photographic Process."

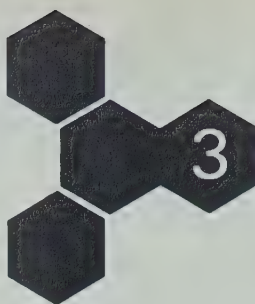
ADDITIONAL INVESTIGATION

Devise an experiment to determine the composition of the residue in beaker #2.

The student may see two kinds of crystals and guess that two substances are present. He may think to test a sample with silver nitrate solution to see if there is chloride present.

Perhaps a more valuable extension is to collect quantitative data to show that the NaCl used (reacted with silver in solution) is related to the silver nitrate in a simple mole ratio (1/1).

Principles of Chemical Reactions



The student begins to learn the part molecules take in chemical reactions. Formulas of compounds start to have more meaning and stoichiometry is introduced. The previous experiments are augmented by a partial formula determination.

Schedule of Experimental Work

TEXT SECTION	EXPERIMENTAL WORK	FOR DETAILS SEE PAGE
3-1	Demo. 9	43
3-2 to 3-6	Expt. 9 (any time)	45

DEMONSTRATION 9

MODELS FOR SHOWING EQUATION BALANCING

PURPOSE

This is an important demonstration. It provides an excellent introduction to ideas of conservation of atoms and balance of equations. It gives a visual basis to these concepts that is difficult to achieve in other ways. With the use of models the level of abstraction is reduced considerably.

TIMING

Early in Section 3-1. Five minutes of class time required.

MATERIALS NEEDED

- 6 Styrofoam spheres, 2 inch diameter
- 3 Styrofoam spheres, $2\frac{1}{2}$ inch diameter
- 3 pieces of chenille stem, $1\frac{1}{2}$ inch long
- some white glue
- paint, white latex for undercoat and white and red enamel for final coat
- calipers, hacksaw blade, garnet paper

PRODUCTION OF MODELS

Use the general directions given in Demonstration 7, p. 26. Specifically, roll the 2 inch spheres to 4.4 cm diameter and the $2\frac{1}{2}$ inch spheres to 5.6 cm diameter. Paint them with the white undercoat, and later add a coat of white enamel to the smaller ones (hydrogen) and red enamel to the larger ones (oxygen). Cut the spheres according to the drawings in Figure 5. Dig out two of the hydrogen atom models so they can eventually fit on the oxygen sphere. Assemble one H_2O model permanently glued, and two H_2 plus one O_2 by means of the stems. Mark and punch a radial hole in each oxygen sphere so that the hole makes an angle of 105° to the chenille stem in the flat surface.

CLASSROOM PRESENTATION

Show each model and identify them as oxygen, hydrogen, and water molecules. Explain that in the gas phase the first two are in rapid motion, colliding with each other and, with the right conditions, reacting to form water. Demonstrate the motion and collision with a model in each hand. Show that many collisions are not fruitful; the molecules recoil without reacting. When a successful collision occurs, the two molecules break apart and the two hydrogen atoms bond to one oxygen. Show this with the models. Then draw attention to the remaining oxygen atom model. Stress that experimentally this species, atomic oxygen, is not found in any appreciable amount. Get the class to suggest that it may react with another hydrogen molecule. Carry out this reaction with the models. Conclude the demonstration by showing on the chalkboard what has just been shown with the models. Emphasize that the equation is

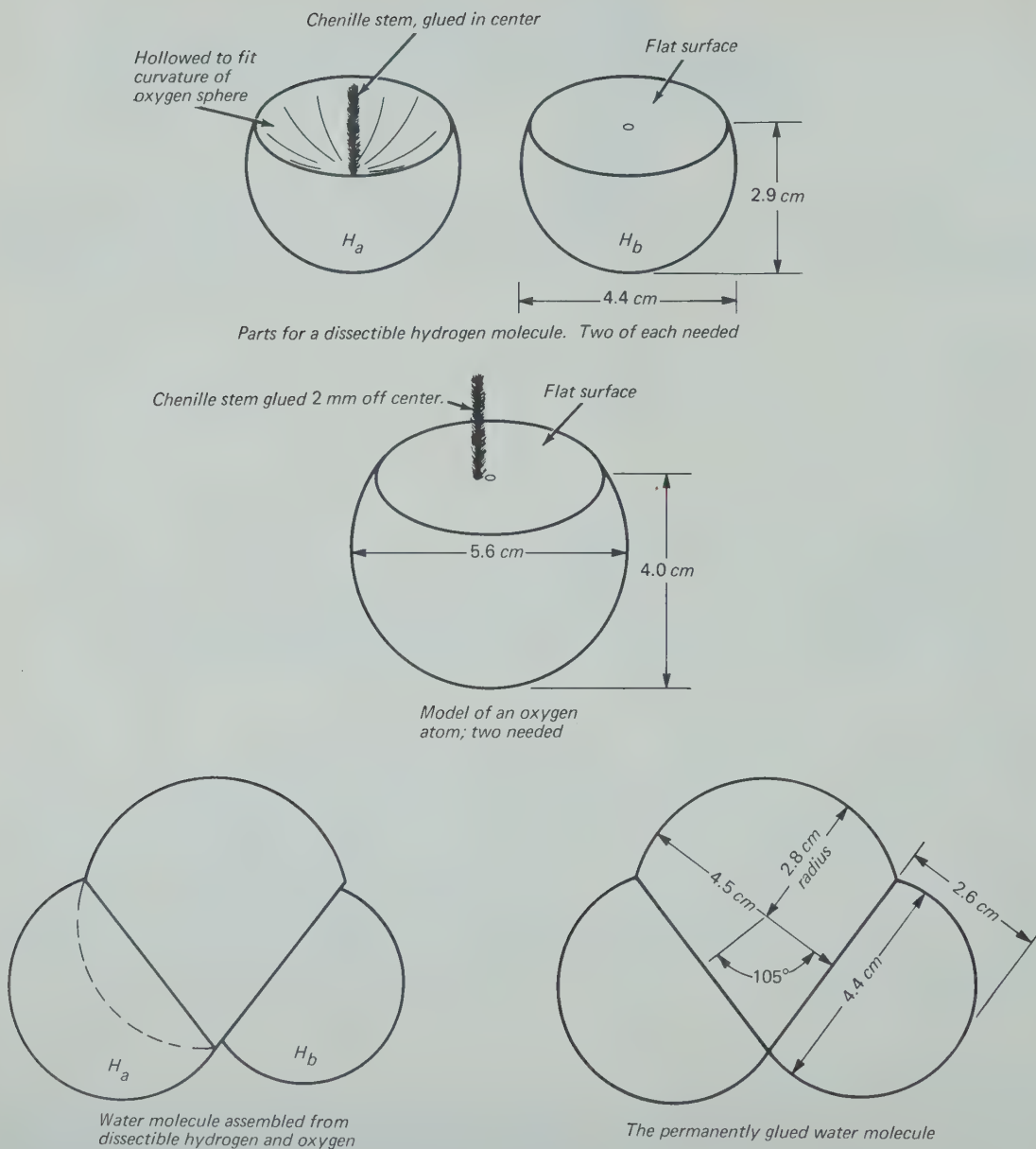
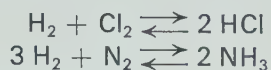


Figure 5
Construction details for dissectible H_2 and O_2 models.

meant to recall for them the colliding molecules and the relative numbers involved in the reaction. Show why we cannot write just one oxygen plus one hydrogen gives one water.

The H_2O assembled in class will not look quite the same as that permanently glued together. The reason is that the bond lengths in H_2 and O_2 are different from those in H_2O . We cannot make models which are correct in both cases.

The same technique can be used with other examples such as



Use the data in Demonstration 7 for making the needed models.

Experiment 9

The Formula of a Hydrate

PURPOSE AND SUMMARY

To enable the student to see that the formula of a compound is based on experimental evidence. The mole concept is reinforced. Experience is gained in quantitative analysis.

A hydrate is heated in a crucible until the water is driven off. From the molar mass of the anhydrous salt, the student can then calculate the moles of water per one mole of salt. See Lab Hint 1.

TIMING

During Chapter 3. This experiment can be done in several short periods of time. The following table gives the time required for the various steps. If the lab time is allotted at the end of a period, the students will hurry, and cooling can take place overnight.

Step	Time (minutes)
a	before class
b	10
c, d, and e	20
f and through the heating in g	10
g (weighing)	5

MATERIALS NEEDED

per student

burner	crucible tongs	ring stand and ring	desiccator (see Lab Hint 2)
crucible and cover	triangle	balance (centigram)	5 g of a hydrate

Suitable hydrates are :

$\text{BaCl}_2 \cdot 2 \text{H}_2\text{O}$	$\text{ZnSO}_4 \cdot 7 \text{H}_2\text{O}$
$\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$	$\text{CaSO}_4 \cdot 2 \text{H}_2\text{O}$ (gypsum)
$\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$	$\text{K}_2\text{CO}_3 \cdot 1\frac{1}{2} \text{H}_2\text{O}$
$\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$	$\text{MnSO}_4 \cdot \text{H}_2\text{O}$

Some of the common hydrates decompose to the oxide at too low a temperature for use in this experiment. An experiment which shows that $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ decomposes in two steps is described in *J. Chem. Ed.*, March 1956, pp. 103–7.

LAB HINTS

1. It is suggested that the mass of one mole of the anhydrous salt, and not its name, be given to the student at first. He should then calculate the ratio of moles of anhydrous salt to moles of water and use these to find a simple numerical relation for the formula of the hydrate.
2. A simple desiccator can be made from a wide-mouthed peanut butter jar having a good seal. A triangle with wires bent downward like a three-legged stool is placed inside with some anhydrous calcium chloride or other dehydrating agent. If the legs of the triangle are too long, cut them with a pair of side-cutting pliers.

PRELAB DISCUSSION

Emphasize that this experiment is typical of those performed by chemists when determining the formula of a compound. If the arithmetic is too much for some of your students, help them through a set of sample data for a hydrate that will not be used in the experiment. For example, 4.95 g of hydrate ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) lost 0.79 g of water. The molar mass of the anhydrous salt is 190 g.

Weighing hot crucibles should be avoided as the convection currents cause errors.

PRECAUTIONS

Observe the usual lab precautions.

POSTLAB DISCUSSION

SAMPLE DATA

This can evolve from a discussion of the corrected student write-ups. The following values are used.

4.84 g hydrate
2.14 g water lost
161 g anhydrous salt/mole

RESULTS OF PROCESSING THE DATA

- 1) Calculate the number of moles of the anhydrous salt you prepared.
Moles of anhydrous salt produced

$$\frac{4.84 - 2.14}{161} = \frac{2.70 \text{ g anhydrous salt}}{161 \text{ g anhydrous salt/mole}} = 0.0168 \text{ mole}$$

Emphasize that to report 0.017 mole is throwing away useful information. If 0.0168 appears to contain 4 significant figures, point out this could be written 1.68×10^{-2} mole. Since the zero is dropped, it cannot be significant.

- 2) Calculate the number of moles of water driven from your sample of hydrate.

Moles of water removed from hydrate

$$\frac{2.14 \text{ g water}}{18.0 \text{ g water/mole}} = 0.119 \text{ mole}$$

Again three significant figures should be given.

- 3) Calculate the moles of water per one mole of anhydrous salt.

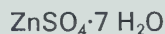
$$\frac{\text{moles water}}{\text{moles anhydrous salt}} = \frac{0.119}{0.0168} = 7.08 \text{ moles H}_2\text{O/mole anhydrous salt}$$

- 4) Write the empirical formula for the hydrate.

The empirical formula will be



or



- 5) Can you suggest reasons why the above method might not be suitable for all hydrates?

Some hydrates may decompose, giving off other gases at temperatures used to drive off the water of hydration; others may require too high a temperature to drive off their water.

The Gas Phase



The particle model is tested further in this chapter. The gas laws are deduced by experiment leading to absolute temperature. The experiments aid by letting the student observe the effect of temperature on gas volume and molar volume. The partial pressure concept allows correction for water pressure.

Schedule of Experimental Work

TEXT SECTION	EXPERIMENTAL WORK	FOR DETAILS SEE PAGE
	Expt. 10	49
4-1, 4-2	Demo. 10	52
4-3	Demo. 11	54
4-4 to 4-6	Demo. 12	56
	Demo. 13	57
	Expt. 11	58
	Demo. 14	61
4-7		
		(any time after 4-2)
4-8	Demo. 15	64

Experiment 10

Temperature-Volume Relation

PURPOSE AND SUMMARY

To let the student discover the mathematical relation between temperature and volume.

The student uses a capillary tube with a mercury plug trapping some air to obtain temperature-volume data. Volume data are obtained by measuring the length of the trapped air column using the degree scale on the thermometer. The data are plotted and extrapolated to determine the temperature for the hypothetical zero volume of the air.

An additional (optional) activity provides opportunity to obtain P - V data using the same capillary tube.

TIMING

Before Section 4-2.

One laboratory period should be enough time. Most students should be able to finish and have time also for the optional experiment.

MATERIALS NEEDED

per individual or pair

- 1 thin wall capillary tube with mercury plug (see Lab Hint 1)
- 2 thermometers, -20° to 150°C
- 1 grease pencil or glass-marking pen
- 1 small rubber band (see Lab Hint 2)
- 1 test tube, 25×200 mm (for hot water bath)
- 1 burner, ring stand, ring, and wire gauze

per class

- plastic tape, several rolls
- crushed ice (see Lab Hint 3)

LAB HINTS

1. Preparation of the capillary tubes.
 - a. Obtain melting point capillary tubes about 20 cm long and 0.8 or 1.0 mm internal diameter.
 - b. Close one end of each tube by heating it in a burner flame. Hold the tube nearly vertical to avoid a bent end.
 - c. Place all the tubes, open ends together, in a bundle and put them in an oven. Make twice as many tubes as will be needed for one class in order to have extras. Heat the tubes to about 250°C to dry and expand the air in them. This requires at least $\frac{1}{2}$ hour.
 - d. Remove the tubes from the hot oven and quickly plunge them, open end first, into a dish of clean, dry mercury. As the tubes cool, mercury will be forced into them. Remove

the tubes when the mercury plug is about 3 cm long. Store the tubes in a stoppered test tube, containing Drierite, until they are needed.

2. Small rubber bands can be cut from soft rubber tubing.
3. Crushed ice baths can be set up in Styrofoam buckets with lids and can be used all day. Have several buckets placed around the lab in convenient locations.

PRELAB DISCUSSION

Briefly discuss the questions that are raised in the introductory paragraphs. Set up a display of the apparatus to be used in the experiment. Tell the students how the capillary tubes, containing the mercury plugs, were prepared. See Lab Hint 1. Discuss briefly the details involved in determining the volume of the trapped air in the tube (steps a and b, Lab Manual p. 29).

PRECAUTIONS

Tell the students to handle the capillary tubes gently. They are very fragile. Should any be dropped and mercury spilled, be sure to pick up all the mercury you can, and sprinkle flowers of sulfur around to react with tiny droplets that may have been overlooked.

Caution the students not to shake or jar the tubes since this may cause the mercury plug to separate into several pieces. When the students finish with their experiments, they should carefully remove the capillary tube from the thermometer and gently return it to a designated container for use in other classes.

POSTLAB DISCUSSION

Discuss the graphs and the significance of the intersection of the extrapolated curve with the temperature axis. (See item 3 under Results of Processing the Data.)

Discuss reasons for the widespread variation in the values obtained for this horizontal axis intercept. You may wish to make a histogram of these values to determine a central tendency.

Discuss the additional activity.

SAMPLE DATA

Temperature, °C	length of air (l_1)	length of air (l_2)
0.0	73.0	17.3
22.6	73.0	25.0
100.0	73.0	49.5

RESULTS OF PROCESSING THE DATA

- 1) Determine the total length of the air column ($l_1 + l_2$) for each temperature.

$$73.0 + 17.3 = 90.3$$

$$73.0 + 25.0 = 98.0$$

$$73.0 + 49.5 = 122.5$$

- 2) Plot the volumes and temperatures on a graph.

See Figure 6.

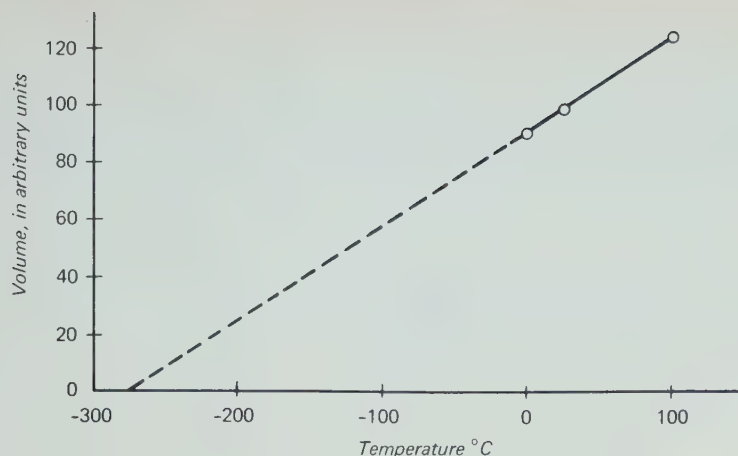


Figure 6
The temperature-volume
relation of dry air.

- 3) By *extrapolation* determine the temperature at which the line intersects the horizontal axis.

−280°C (theoretical = −273°C)

Give reasons why a gas does not behave as suggested by the extrapolated line.

Before any gas reaches this temperature it would have changed into a liquid or solid. Since the molecules of a gas occupy space, it is impossible for a sample of matter to occupy zero volume.

- 4) Express the temperature-volume relation as a mathematical expression.

The volume is proportional to temperature. $T = k_1V - 280$, or $k_2(T + 280) = V$

The value 280 will depend on the student's extrapolation.

SAMPLE DATA (Additional Activity)

Temperature = 22.0°C

Barometric pressure = 755 mm Hg

Position	length of air column	length of mercury plug
horizontal	94.0 vol units	31.0 mm
open end up	90.5 vol units	31.0 mm
open end down	97.5 vol units	31.0 mm

RESULTS OF PROCESSING THE DATA (Additional Activity)

- 1) Determine the pressure of the air in the tube in each of the three different positions.

horizontal, $P = 755$ mm Hg

open end up, $P = 755 + 31 = 786$ mm Hg

open end down, $P = 755 - 31 = 724$ mm Hg

- 2) Calculate the PV product for each different position of the capillary tube.

horizontal, $PV = 94.0 \times 755 = 7.10 \times 10^4$

open end up, $PV = 90.5 \times 786 = 7.11 \times 10^4$

open end down, $PV = 97.5 \times 724 = 7.06 \times 10^4$

3) What effect would increasing the temperature have on the value for the PV product?

Increasing the temperature would cause the volume of air to become larger. The pressures would remain the same. Thus the PV products would be larger but constant for any given temperature.

DEMONSTRATION 10

MOLECULAR MOTION

PURPOSE

To illustrate the kinetic theory (particles in motion), to show that diffusion is a relatively slow process in air, and to convincingly demonstrate that molecules move rapidly when not impeded.

TIMING

During Section 4-1. About 20 minutes needed.

MATERIALS NEEDED

2 glass tubes, see Lab Hint 1
2 bromine bombs, see Lab Hint 2
some NaOH, 1 *M*
aspirator
ring stand, clamp
pinch clamp
glycerol

PREPARATION HINTS

1. Prepare 2 tubes as shown in Figure 7. The numbers refer to the comments below.

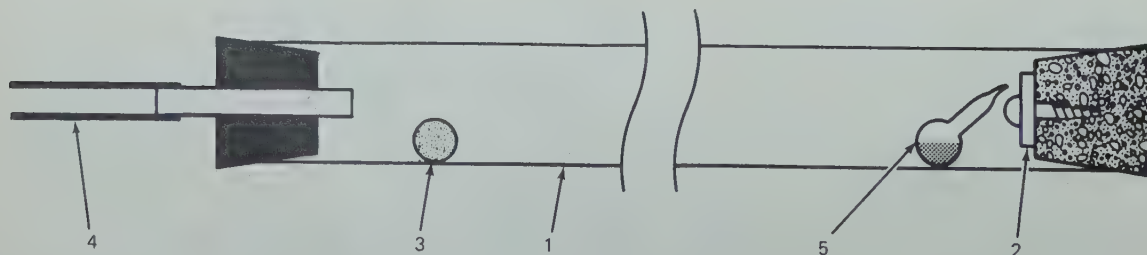


Figure 7
Tube for demonstrating
velocity of molecules.

- 1) a glass tube about 30 mm inside diameter and 4 feet long.
 - 2) a heavy metal washer or piece of lead to help break the bromine bomb. Screw the metal to a cork with a long wood screw and seal the cork in the tube with melted wax.
 - 3) a $\frac{1}{2}$ inch ball bearing or rounded chunk of lead to break the bromine bomb.
 - 4) stiff-wall rubber tubing over glass tube in stopper. One tube uses a plain solid stopper.
 - 5) bromine bomb
2. The bromine bombs can be made in this way. Cut several one foot lengths of 6 mm soft glass tubing. Fire polish one end and seal the other closed. Blow a $\frac{1}{2}$ inch bubble in the closed end, then draw out the other end as in Figure 8. The inside diameter of the drawn-

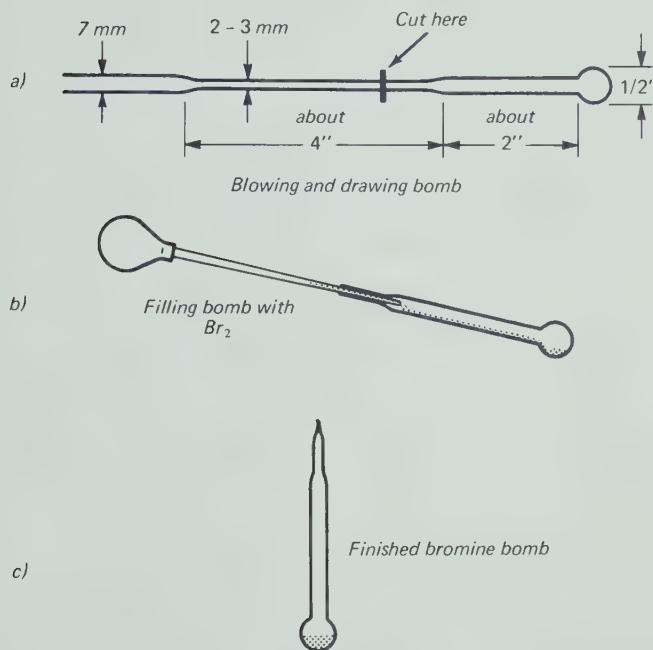


Figure 8
Steps in making bromine bombs.

- out tube must accept a drawn-out medicine dropper. Then break the tube about 1 cm from the wide section. The long piece of tubing can now have another bubble blown in it. Prepare 30–40 units as shown at the top in Figure 8. Make a drawn-out medicine dropper.
3. To fill the bombs, move to a hood or into a well-ventilated place with the wind from behind you. Bromine burns are dangerous. Should any get on your skin immediately rub glycerol on the area. Do NOT BREATHE the vapors. Have a lit burner nearby and wear safety glasses. Pick up a bomb with a pad of wet towelling. Get some Br_2 in the dropper. Insert tip of dropper into the bomb and inject a few drops of the liquid. Withdraw the dropper and seal the end of the neck in the flame. Put the filled bomb aside in a position so the Br_2 cannot come in contact with the hot end.
 4. Fill many bombs in this way and leave them in the hood overnight. Those with leaks will be empty by morning. Store the good ones between layers of cotton in wide-mouth jars. Wax the inside of the lid. A supply for several years can be made in an hour or two.

CLASSROOM PRESENTATION

Exhibit a pair of the bombs. Explain what is to be done. One will be broken in the evacuated glass tube, the other in a tube which is full of air. Have the students decide which bomb is to be used for the first part. Then put the bomb carefully in the sealed bottom of a tube. Keep the end containing the bomb slightly higher than the open end while you insert the metal ball and stopper.

Invert the tube to drop the ball on the bomb. Repeat until the bomb breaks, then fasten the tube vertically to ring stand. Note the time and mark the top of the Br_2 level. Discuss the rest of the demonstration and check in 5 minutes. Diffusion is slow when air is present.

For the evacuated test, load a bromine bomb as before and put the ball in. Connect the aspirator to the tube in the stopper and evacuate for a few minutes. Explain that the tube will be sealed off from the aspirator, and inverted to break the bomb. Prime the students to count the number of seconds the colored gas requires to reach the top of the tube. Double the evacuation hose and close it with a pinch clamp. On the count of three, invert the tube. The gas will travel to the top instantly. If the Br_2 moves only part way up the tube, either air leaked in or the aspirator is not functioning properly.

This demonstration is quite dramatic and convinces most students that molecules of gas do move. Actually, bromine molecules move relatively slowly because they are so heavy. At 0°C and 1 atm the average velocity is about 400 miles/hr. Hydrogen molecules move at 3600 miles/hr or nearly 10 times as fast.

Finally, call attention to the diffusion in the tube containing air. Lead the students to propose that the slowness is the result of the presence of other molecules. You may wish to suggest that if this is the explanation, then as air is admitted to the full tube of bromine the reddish gas should be pushed down. Admit air carefully and observe the decrease in volume of bromine.

The bromine can be safely removed by adding 10–20 cc of 1 *M* NaOH to each tube. Swish the liquid around until the bromine color disappears.

DEMONSTRATION 11

PRESSURE-TEMPERATURE RELATION

PURPOSE

To show the P - T relation for a gas and to introduce the absolute temperature scale.

TIMING

This demonstration should be done near the start of Section 4-3.

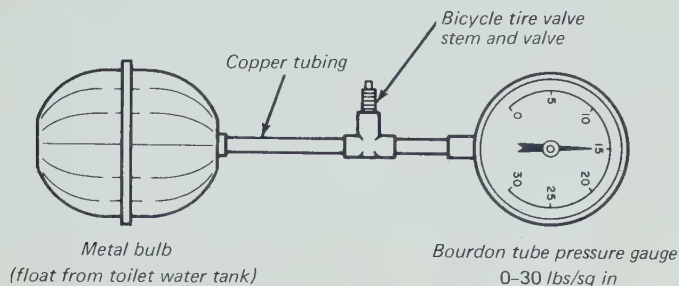
MATERIALS NEEDED

1 apparatus as described below under *Lab Hint*
3 beakers, 2-liter, or large battery jars
ice
Dry Ice

acetone or amyl alcohol
burner
ring stand
ring and wire gauze

LAB HINT

The apparatus can be constructed as shown in this drawing :



All connections must be made airtight. The bulb must be soldered around the seam to make it airtight. The gauge should be set to read atmospheric pressure with dry air in the bulb at atmospheric pressure. The apparatus is listed as John's Apparatus in the tabulation at the back of this Teacher's Guide.

CLASSROOM PRESENTATION

1. Prepare these three temperature baths :
boiling water,
ice-water mixture,
Dry Ice-amyl alcohol or Dry Ice-acetone mixture.

The bath containing Dry Ice will be at about -78°C . The liquid acts as a heat-conducting medium, but the temperature is determined by the $\text{CO}_2(\text{s})$. The alcohol gives a bath that rarely froths or "bumps," is less of a fire hazard, and stays mushy better than one made with acetone. The alcohol has a stronger odor than the acetone.

2. Record room temperature and gauge pressure.
3. Obtain gauge pressure readings for each of the three other temperatures by immersing the bulb completely in each of the baths. Read the pressure gauge with as much care as you can.

STUDENT ASSIGNMENT

1. Have the students plot the data with temperature along the abscissa (horizontal) and gauge pressure along the ordinate (vertical). The origin should be at $t = -300^{\circ}\text{C}$ and $P = 0$.
2. Have the students extrapolate the line formed by the four measured points to the line $P = 0$.

SAMPLE DATA

Figure 9 gives an example of the expected results.

1. Determine what fraction the pressure change (ΔP) per degree Celsius is of the pressure at 0°C ($P_{0^{\circ}\text{C}}$). Express the fraction with a numerator of one. As an example, for the temperature range $0-100^{\circ}\text{C}$,

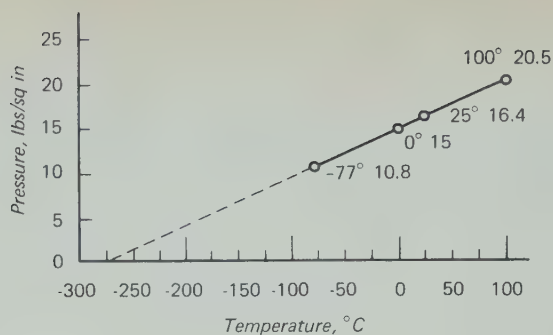


Figure 9
Pressure-temperature
relation for air.

$$\frac{\text{change in pressure per degree}}{\text{pressure at } 0^{\circ}\text{C}} = \frac{\frac{\Delta P}{t_2 - t_1}}{P_{0^{\circ}\text{C}}} = \frac{\frac{5.5}{100}}{15} = \frac{1}{270}$$

DISCUSSION

The demonstration can help develop the idea of absolute zero. The fractional change in pressure per degree Celsius change in temperature is generally very close to $1/273$. This can be used to "invent" or support the Kelvin temperature scale.

DEMONSTRATION 12

DIFFUSION RATE INCREASES WITH TEMPERATURE

PURPOSE

To show that the rate of diffusion is dependent on temperature and thus, that the molecular motion varies with temperature.

TIMING

During Section 4-4. About 10 minutes are needed early in a period.

MATERIALS NEEDED

2 beakers, 250 ml	burner, ring stand, gauze
ink	ice
medicine dropper	

CLASSROOM PRESENTATION

Boil water in one beaker and cool some in another. Remove the heat source and the ice and let the water become quiescent; 5 minutes. Then add one drop of ink carefully to each beaker. Continue other work while diffusion takes place.

After the greater diffusion rate in the hot water is apparent, develop the idea that a reasonable explanation comes from assuming the molecules are moving faster there than in the cold water.

DEMONSTRATION 13

EFFECT OF EQUATING LIQUID LEVELS IN PRESSURE MEASUREMENT

PURPOSE

To show students the effect of changing the position of a gas measuring tube on the volume of the contained gas.

TIMING

The day before Experiment 11.

MATERIALS NEEDED

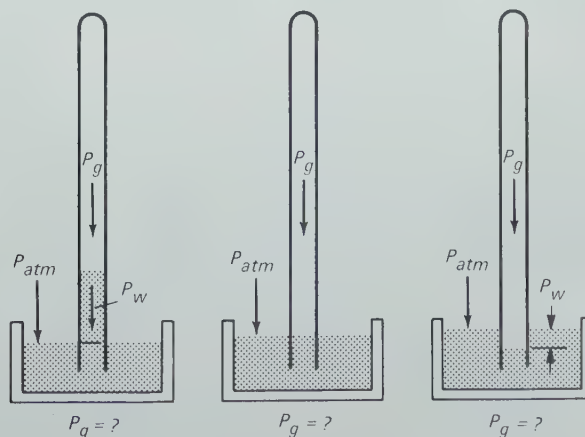
gas-collecting tube
graduated cylinder, 500 ml, or hydrometer jar

CLASSROOM PRESENTATION

- Put about 10 ml of water in the gas-collecting tube. Invert it in the jar of water.
- Have a student read the position of the meniscus inside the tube while the meniscus is:
 - above the water level in the jar
 - near the water level in the jar
 - below the water level in the jar

FOLLOW-UP

Have these drawings on the board the day the experiment is being done. Have the students calculate the pressure values.



ANSWERS

$$P_g = P_{\text{atm}} - P_w$$

$$P_g = P_{\text{atm}}$$

$$P_g = P_{\text{atm}} + P_w$$

Experiment 11

The Reaction of Magnesium with Hydrochloric Acid

PURPOSE AND SUMMARY

To give an experimental basis for calculating the molar volume of hydrogen gas (room temperature, 1 atm) and later (after Section 4-3) at STP. This experiment is traditionally used as an "equivalent weight" determination, and it is important that you reflect on the reasons why we now include it. They are:

- 1) We wish to emphasize the mole concept.
- 2) Using both "equivalent weights" and moles confuses many students. Only one is needed.
- 3) The "equivalent weight" of a substance depends on the reaction in which it is used. For example, the "equivalent weight" of KMnO_4 when used with H_2O_2 in an acidic solution is 158/5 g while if used in a basic solution is 158/3 g. On the other hand, a mole of any substance is always the same amount.

The student determines the mass of a piece of magnesium ribbon by measuring the length of a piece with a given mass/length ratio. He measures the volume of gas formed as the metal reacts with hydrochloric acid in a gas collecting tube. Allowance is made for the vapor pressure of water and the molar volume of hydrogen is determined.

This is a very satisfying experiment, since both the precision and accuracy are high, and the manipulation is simple yet interesting.

TIMING

This experiment should be done after assigning Section 4-1.

About 30 minutes of lab time are needed.

MATERIALS NEEDED

per student or pair

- 1 gas-measuring tube, 50 ml (see Lab Hint 2)
- 1 one-hole stopper (size #0 or #00 to fit measuring tube)
- 1 ring stand and clamp
- 1 beaker, 400 ml
- 1 metric ruler
- 5–6 cm of magnesium ribbon (see Lab Hint 1)
- 18 inches #30 bare copper wire
- 10 ml of 6 M hydrochloric acid (513 ml conc. reagent/liter)

per class

- 1 thermometer
- 1 barometer (see Lab Hint 4)
- 10 water reservoirs (see Lab Hint 3)
- 10 plastic squeeze bottles of water

LAB HINTS

1. The magnesium ribbon, if free of corrosion, is very uniform. If the package has been opened previously, the outermost section should be discarded in order that an uncorroded sample is obtained. Weigh the ribbon in 1 meter lengths (about 1 g) in order that the precision on the ± 0.01 gram balance will be 1 part in 100. If the ribbon is weighed on an analytical balance the precision is 1 part in 10,000, which, although unnecessary, may be useful if you wish to use the data from this experiment for further discussion of precision. The student is asked to measure the length of the ribbon to one part in 100, or ± 0.5 mm, in his 5 cm strip, and this is certainly the least precise measurement. He should be reminded to observe the finite width of the ruler lines.

It is wise for you to check the volume of H_2 from a given length of Mg either experimentally or by calculation, since it is desirable to collect as large a volume as can be measured in a single tube and because temperature and pressure differences make it impossible to predict the optimum length of Mg for every local set of conditions. At high altitudes a piece as short as 4 cm may be used.

2. If gas-measuring tubes are not available, it is possible to calibrate the portion of a buret between the 50.0 ml mark and the valve, but there is some difficulty in reading the markings. An unmarked 10 mm (or more) diameter glass tube, closed or stoppered, may be used and the volume measured later in a graduated cylinder. The gas-measuring tube marked to 0.1 ml divisions gives an uncertainty of about ± 0.05 or $\pm 0.1\%$ for 50 ml of hydrogen.
3. A battery jar or large beaker (2 or 3 liter size) is ideal. Hydrometer cylinders are excellent also.
4. The degree of precision from other parts certainly does not warrant using the vernier scale in recording the barometer reading, but this may be done for instructional purposes and to give the students another "choice" when doing the final calculations. Nor does one need to use corrections for the temperature, latitude, etc. A correction of 2–3 mm in 700 mm is only 1 part in 200 or 300, and therefore more precise than some other measurements being used.

PRELAB DISCUSSION

It is important that the student have an understanding of the various steps in this experiment. Since each of the measurements involved may be done with considerable precision, he should focus his attention on working carefully. Tell how the magnesium ribbon has been weighed, and point out the necessity for leaving square corners when a small piece is cut off. Discuss how the difference in density of the HCl solution and the water permits a water column to rest on top of the acid. Discuss the reason for adjusting the level of the water inside and outside the tube before reading the volume. See Demonstration 13.

PRECAUTIONS

Observe the usual lab precautions.

POSTLAB DISCUSSION

As previously stated, the precision of all the measurements in this experiment is high. If, when the results of the individuals in a class are compared, there is a trend—for example, if most of the individual results are low but in agreement with each other—the most likely source of error lies in the weighing of the Mg ribbon, and this should be rechecked.

Since the calculations are somewhat involved, you may wish to de-emphasize considerations of uncertainty until you collect data for a class discussion.

SAMPLE DATA

Mass of Mg ribbon	0.825 g/m	($\pm 0.06\%$)
(Mass of Mg ± 0.0001 g $\sim 0.01\%$; length of meter of Mg ± 0.0005 m $\sim 0.05\%$)		
Length of Mg ribbon used	5.50 ± 0.05 cm	($\pm 1\%$)
Volume of hydrogen collected	47.5 ± 0.05 ml	($\pm 0.1\%$)
Temperature of water	$25.0 \pm 0.2^\circ\text{C}$	($\pm 1\%$)
Temperature of room	$25.0 \pm 0.2^\circ\text{C}$	($\pm 1\%$)
Barometer reading (room pressure)	758 ± 2 to 3 mm	($\sim 0.3\%$)
Vapor pressure of water	23.7 ± 0.2 mm	($\sim 1\%$)

The student has no basis for estimating the lack of precision in the vapor pressure value other than his knowledge of the uncertainty in the temperature. From the handbook one may estimate that a temperature range of $\pm 0.2^\circ\text{C}$ would give an uncertainty of about $\pm 1\%$. For example, at 24.8°C , the vapor pressure is 23.5 mm; at 25.2°C , it is 24.0 mm.

RESULTS OF PROCESSING DATA

1. Determine the mass of the magnesium you used, knowing the grams per meter and the length of your piece of ribbon.

Mass of Mg ribbon:

$$(0.825 \text{ g/m} \pm 0.06\%) \times (5.50 \text{ cm} \pm 1\%) \times 10^{-2} \text{ m/cm} = 4.54 \times 10^{-2} \text{ g} \pm 1\%$$

2. Determine the number of moles of magnesium used.

$$\frac{4.54 \times 10^{-2} \text{ g Mg} \pm 1\%}{24.3 \text{ g/mole Mg}} = 1.87 \times 10^{-3} \text{ mole Mg} \pm 1\%$$

3. Determine the partial pressure of the hydrogen gas. (Question condensed.)

$$\begin{aligned} P_{\text{H}_2} + P_{\text{H}_2\text{O}} &= P_{\text{room}} \\ P_{\text{H}_2} &= P_{\text{room}} - P_{\text{H}_2\text{O}} \\ P_{\text{H}_2} &= 758 - 24 = 734 \text{ mm} \pm 1\% \end{aligned}$$

4. Determine the volume the hydrogen gas would have at one atmosphere pressure (760 mm). (Question condensed.)

$$\begin{aligned} V_{\text{new}} &= V_{\text{measured}} \left(\frac{P_{\text{H}_2}}{760} \right) \\ &= 47.5 \times \frac{734}{760} = 45.9 \text{ ml} \pm 1\% \end{aligned}$$

5. Calculate the volume of dry hydrogen which would be produced by one mole of magnesium at room temperature and one atmosphere pressure.

Since 1.87×10^{-3} mole Mg $\pm 1\%$ produced 45.9 ml $\pm 1\%$ H₂,

$$\frac{45.9 \text{ ml} \pm 1\%}{1.87 \times 10^{-3} \text{ mole} \pm 1\%} = 24.5 \times 10^3 \text{ ml/mole} \pm 1\%$$

$$= (24.5 \text{ liters} \pm 0.2 \text{ liter})/\text{mole}$$

6. Given that one mole of Mg produces one mole of hydrogen, H₂, what is the volume of one mole of hydrogen at room temperature and 1 atmosphere pressure?

If one mole of Mg produces one mole of H₂ and, if 1 mole of Mg produces 24.5 liters H₂, then one mole of H₂ occupies 24.5 liters at room temperature and one atm.

7. If one mole of hydrogen weighs 2.0 g, what is the mass of a liter (the density) of hydrogen at room temperature and one atmosphere pressure?

At one atmosphere and 25°C, 24.5 liters of H₂ contain one mole of H₂. If one mole weighs 2.0 g, then

$$\frac{2.0 \text{ g}}{24.5 \text{ liter}} = 0.082 \text{ g/liter of H}_2 \text{ at 1 atm and 25°C}$$

ADDITIONAL INVESTIGATION

Most metals react very slowly. Aluminum and zinc are satisfactory; iron requires overnight. See Demonstration 10.

DEMONSTRATION 14 (after Experiment 11)

REACTION OF Al, Mg, AND Na TO PRODUCE H₂— MOLAR RELATIONS FROM THE GAS VOLUME PRODUCED

PURPOSE

To show that (1) one mole of hydrogen is *not* produced by one mole of every metal reacting with acid, (2) sodium is a bright shiny metal, and (3) the coefficients in equations are based on experiments.

By reacting Na, Mg, and Al with HCl and calculating the moles H₂/mole metal these goals are attained.

TIMING

Shortly after Experiment 11.

MATERIALS NEEDED

3 cylinders, (25 × 200 mm test tubes are barely satisfactory. 250 ml hydrometer cylinders are excellent.)

1 container for holding the 3 cylinders

A variety of clamps and ring stands for holding cylinders vertical in container.

150 ml, 6 M HCl (conc. HCl diluted 1/1 with water)

stoppers for cylinder used with Al

several feet of thin copper wire

1 foot of stiff copper wire

samples of each metal

Al About 0.074 g (2.7 millimoles) for each 100 ml H₂ desired. If Al foil is used, determine the mass for several hundred cm², and calculate cm²/0.074 gram. One millimole of Al is 0.0269 gram.

Mg One millimole is 0.0234 gram. Each 2.7 millimoles used will produce about 67 ml H₂.

Na One millimole is 0.0230 gram. Each 2.7 millimoles used will produce about 33 ml H₂.

See the technique below for getting Na metal into tubes and for calculating the mass. It may be convenient to estimate the Na for a trial run, measure the amount of H₂ gas produced, and use this to calculate how much the Na weighs per centimeter of tubing. Water may be substituted for dilute HCl, but if the sodium is encased in 6–8 mm tubing, be prepared for a slow reaction, since the H₂ gets trapped in the tube. A wire attached to the tube of Na enables one to jiggle it and dislodge bubbles.

PREPARING SODIUM METAL IN GLASS TUBES

The general method consists in letting the atmosphere push the molten sodium into a warm glass tube, where it solidifies. The tubes can then be cut and stored.

1. Have readily accessible:

a) About 6 pieces of 6 mm soft glass tubing, each about 30 cm long. 5 mm tubing might work, but experience with 4 mm tubing shows that the bore is so small as to retard the entrance of water. This is a disadvantage if you wish to demonstrate the reaction of sodium with water.

b) About 20 cm³ of sodium.

c) A crucible or test tube to hold the sodium.

d) A vacuum source with a control valve (a screw-type pinch clamp is okay). *Do not use an aspirator.*

2. Thoroughly dry the work place. Put on a pair of safety glasses—do not work alone. Hold a piece of sodium with tongs, and cut off the crust. Use paper towels to blot most of the oil off the sodium. If this is not done, an emulsion will form when the sodium is melted, making it virtually impossible to get a tube full of bright, solid metal.

3. Very gently warm the sodium in a crucible or test tube until melted (97.5°C). Remove the flame.

4. Attach the vacuum line (turned off) to a piece of 6 mm soft glass tubing, and warm the glass gently. If the tube is cold, the sodium will soon solidify, preventing complete filling of the tube.

5. Put the free end of the glass tube in the molten sodium, and slowly turn on the vacuum until sodium is pushed nearly to the top of the tube. Pinch off the vacuum, but keep the end of the

- tube under the sodium surface until that in the tube solidifies. If removed too soon, the molten sodium will run out of the tube, leaving a hollow pipe that will tarnish in a few months.
6. Repeat step (5) to fill as many tubes as desired.
 7. These tubes can be cut and stored under kerosene or toluene. Toluene is better, since it absorbs less water. Nick the glass with a file, hold with toweling, and snap. The sodium may be cut with a knife, or merely twisted until separated.
 8. Unused, melted sodium may be poured on a dry surface and, when solidified, returned to a storage bottle. The crucible or test tube should be cleaned, and the scrap sodium disposed of by allowing it to react with small quantities of methanol. When the addition of more methanol causes no reaction, and when *all* solid material has reacted, pour the solution down the drain.
 9. To obtain a specific mass of sodium metal, determine the volume of the tube. This can be done roughly by using linear measurements, or more precisely, by filling a known length of tube with mercury. The mass of mercury used, divided by its density (13.5 g/ml) will give its volume. The volume per centimeter multiplied by the density of sodium (0.97 g/ml) will give grams of Na per centimeter of tube.

CLASSROOM PRESENTATION

Have cylinders for Al, Mg, and Na filled with water floating on 75, 25, and 5 ml of 6 M HCl in the respective cylinders. Also have tank $\frac{2}{3}$ full of water.

- 1) Wrap the Al in copper wire. Insert it into the water of the cylinder. Insert stopper with several small holes, or use a glass cover, then invert the cylinder in tank of water. Clamp the cylinder in position so that the acid cannot fall away from the Al too fast.
- 2) Prepare the Mg in a similar manner. Since H_2 is generated fairly rapidly, the cylinder should be closed with a large hole stopper, or a glass plate should be used in the transfer and the end of the cylinder left open but close to the bottom of the tank.
- 3) Since Na reacts with water, setting up its cylinder has to be done carefully. Cover the cylinder with a glass plate and invert it in the tank of water. Remove the glass plate. Attach a stiff copper wire to the glass tube containing Na. Lift the cylinder so that its open end is about 1 cm below the water level in the tank. Quickly manipulate the wire so that the Na is put inside the cylinder.

FOLLOW-UP

Have the students write balanced equations for the reactions using two moles of each metal. The one, two, and three moles of hydrogen correspond to the observed volumes.

DEMONSTRATION 15
LIQUID TO GAS CHANGE OF PHASE

PURPOSE

To help the students see the great difference in volume between a liquid and a gas. To give the students experience with liquid to gas transition using a low boiling temperature substance.

TIMING

Can be used with Section 4-7 when difference in volume of a gas and liquid is first brought up. Or it could be used later in Chapter 5, Section 5-3.

Method I

MATERIALS NEEDED

- 10 ml liquid trichloromonofluoromethane, CCl_3F , (marketed under trade names, Ucon 11 or Freon 11)
- 1 wide mouth jar, quart, peanut butter type
- 1 freezer bag, clear plastic, quart size
- 15 inches plastic tape, electrician black
- several ice cubes

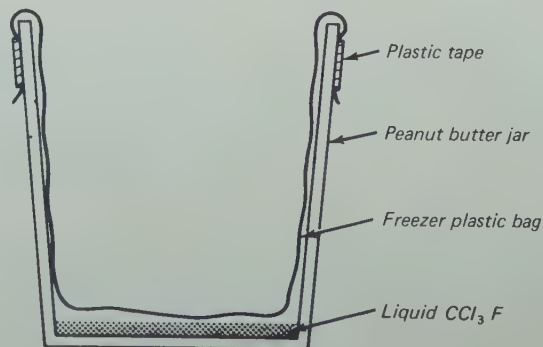
PREPARATION

Have warm water (about 35°C) available in a container large enough to accept the quart jar. Trichloromonofluoromethane boils at 23.8°C , so the water doesn't have to be very warm. Also have several ice cubes available.

CLASSROOM PRESENTATION

Place about 10 ml of liquid CCl_3F into the jar. Place the plastic freezer bag inside the jar and fold the bag out over the mouth of the jar as shown in Figure 10.

Figure 10
Liquid-gas phase change apparatus.



Squeeze as much air out of the jar as possible. Fasten the bag securely to the outside of the jar using plastic tape. Place the jar in a shallow pan of warm water to boil the liquid CCl_3F . The gas as it is produced inflates the bag. As soon as the bag becomes fully extended above the jar remove the jar from the warm water. Hold an ice cube against the top of the bag. Liquid CCl_3F forms and "rains" down inside the bag. The phase change back and forth from liquid to gas and gas to liquid can be repeated several times.

Perform the demonstration without comment except to explain to the students what you are doing and to amplify their observations. Let them make their own observations and ask them to explain the phenomena. Often students think the ice is melting and leaking through the bag. Interesting experiments can be performed to test this hypothesis.

Make a rough comparison of the volumes, liquid to gas.

Method II

MATERIALS NEEDED

1 syringe, 250 ml
10 ml CCl_3F

CLASSROOM PRESENTATION

A similar demonstration can be conducted using a large glass syringe (250 ml). If a syringe is used, lubricate the plunger with a suitable substance (petroleum jelly or stopcock grease). With a hypodermic needle in place, draw a small amount of CCl_3F into the syringe. Hold the syringe vertically with the needle up and expel nearly all of the liquid. Leave just enough in the syringe to cause the plunger to be pushed out to full volume when all the liquid has changed to gas. Try the experiment in advance so that you will know just how much liquid to use. Stick the needle into a solid rubber stopper. Place the syringe into a warm water bath to evaporate the liquid. Place the syringe in a cold water bath to condense the gas. You can force evaporation by pulling out on the plunger to lower the pressure inside the syringe. You can force the gas to condense by pushing in on the syringe to increase the pressure on the vapor.

Liquids and Solids

An Extension of the Kinetic Theory



The kinetic theory of molecular motion is extended to the condensed phases. Phase changes are introduced and form the basis for the laboratory work. The three experiments

- 1) show the constant temperature during phase change,
- 2) begin the study of heat effects, and
- 3) provide some experience with a simple calorimeter.

Schedule of Experimental Work

TEXT SECTION	EXPERIMENTAL WORK	FOR DETAILS SEE PAGE
5-1	Expt. 12	67
	Expt. 13	71
	Expt. 14	73
5-2 to 5-3	Demo. 16	77
5-4	Demo. 17	78
5-5 to 5-6	Demo. 18	79

Experiment 12

Warming and Cooling Behavior of a Pure Substance

PURPOSE AND SUMMARY

To study the characteristic warming and cooling behavior of at least one pure substance, *paradichlorobenzene*, and to determine its melting and freezing temperature. See Lab Hint 4.

The student observes and records time-temperature data for *paradichlorobenzene* as it is cooled and warmed. He makes a graphical study of this characteristic behavior by comparing the cooling curve for *paradichlorobenzene* to the warming curve of the water bath and the warming curve for the *paradichlorobenzene* to the cooling curve of the water bath. The experiment has a real "element of discovery" as the student observes a constant temperature during the changes in phase.

TIMING

Before Section 5-2 in the Textbook.

The experiment requires about 30 to 40 minutes. Some students may have time to investigate the cooling behavior of other solids as suggested in the Additional Investigations section.

MATERIALS NEEDED

per pair

- 15 grams of *paradichlorobenzene* in an 18 × 150 mm test tube. See Lab Hint 1.
- 2 thermometers, -20 to 150°C
- 1 ring stand, ring, wire gauze, and clamp. See Lab Hint 2.
- 1 burner
- 1 beaker, 400 ml

per class

- clock with sweep-second hand

LAB HINTS

1. Tubes containing about 15 grams of *paradichlorobenzene* should be prepared in advance and supplied to the students. Melt the substance in a metal can and pour it into the test tubes. Determine the height that 15 grams will reach by melting this amount in the first tube. Fill the others to the same height.
2. A clamp should be used as a test tube holder, Lab Manual Figure 12-1, in order that the tube of *paradichlorobenzene* may be clamped in place quickly after the solid melts. If heating is done with the test tube in the clamped position, the student is too likely to let the substance overheat at the bottom of the tube. The heating must be done gradually since the temperature rises rapidly as soon as the solid has melted.

3. About 400 ml of hot water (70°C) is needed for each pair of students in the warming part of the experiment. If hot tap water is not available, you may want to start heating some water while the students are doing the first part of the experiment.

PRELAB DISCUSSION

For best results in studying the cooling behavior, the water bath should not be below 30°C to start.

For the warming behavior, the temperature and amount of water have been selected so that the water will have sufficient thermal energy to melt the solid and warm the liquid to about 60°C . Make sure the students fill their beakers four-fifths full and warm the water to between 65 and 70°C . If warmer than this, the flat portion of the curve will be lost; if cooler than this, there will be too little thermal energy and the substance will not reach 60°C .

Students may need additional instruction in reading a thermometer. Some may still be making errors due to parallax.

Some students may need help with the graphical presentation of the data. It may be best to let them make the effort first on their own, making some mistakes, then help them later as the need arises. Some help is available in Appendix 6 of the Laboratory Manual.

You may want to have different pairs of students work with different substances. Some care is needed in the selection of these chemicals. The melting temperature should be 50°C or lower. Some substances tend to undercool too much. This brings up an additional concept which tends to confuse rather than help at this time. See Additional Investigations for suggested suitable chemicals.

The amount and temperature of water needed in the bath should be determined by experiment. Important factors to be considered in working out a procedure are: amount of substance used, the heat of fusion for the substance, and the melting temperature.

PRECAUTIONS

Tell the students to be careful when heating liquids in a test tube. They are not to heat it in a single spot—especially not at the bottom. Some liquids tend to superheat and then suddenly change into gas causing hot liquid to be blown out the tube. Tell them never to aim the test tube toward themselves or others while they are heating it. See Lab Hint 2.

POSTLAB DISCUSSION

Discuss the experiment briefly. Students will note the flat portions of the curves and that they occur at about the same temperatures for heating or cooling. These are the important points to be “discovered.” If some of the students observed similar behavior for other substances you can make the point that the same behavior can be observed for many substances. Compare the shapes of the water bath curves to those of the *paradichlorobenzene*. Experiments 13 and 14 will help answer some of the questions raised in this experiment. Ask the students to speculate concerning their reasons for the flat portions of the curves. Do not try to tell the whole story at this time, rather, wait until they have done the next two experiments.

Show the class some good graphs made by students. Be sure it is understood that plotting data and interpreting graphical data are skills they are expected to develop and use. Discuss criteria used in selecting a suitable scale. Give an explanation about smoothing the curve.

It may be advisable to have some of the students prepare a second graph to correct errors made in their first attempt.

In your discussion, mention the possibility of determining the melting temperature from the graph. The student does not yet have the background for interpreting the energy relation, but will certainly have an opportunity to think. He can see that the water bath was a source of energy and as energy was transferred the temperature of the solid increased until melting started; further, the temperature of the test substance did not rise again until melting was completed. He may come up with the correct conclusion that energy was used to melt the substance. How he expresses this will depend upon his degree of sophistication in science.

Discuss sources of error and uncertainty in measurement. Suggest that they might wonder about the questions that have come up in this experiment and look for answers in subsequent experiments.

SAMPLE DATA

See Figure 11.

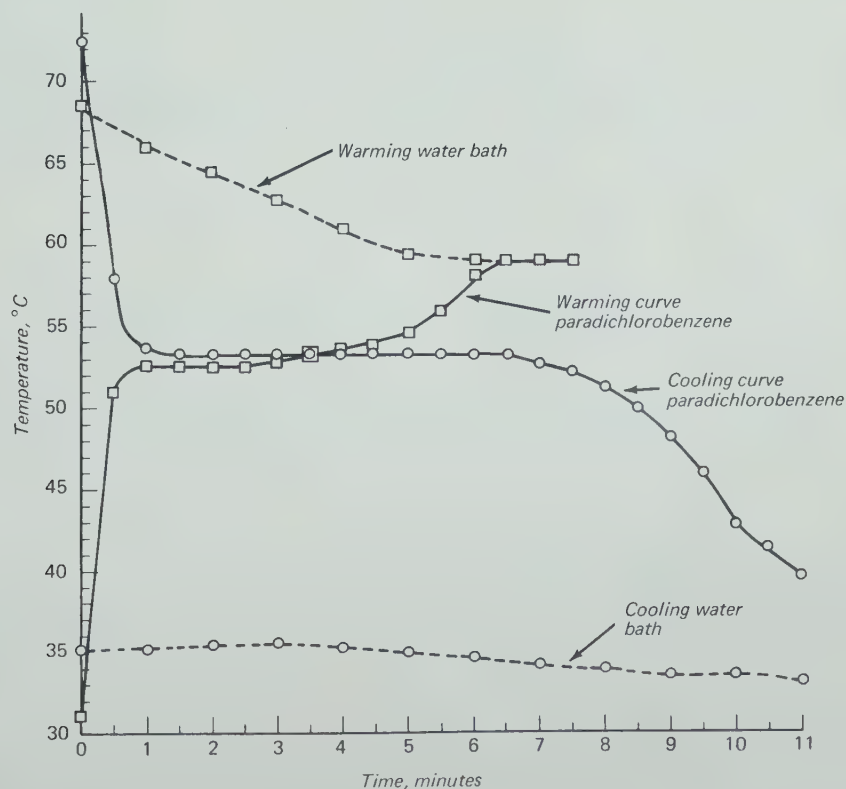


Figure 11

Cooling and warming curves for *paradichlorobenzene*.

RESULTS OF PROCESSING THE DATA

- 1) In the evaluation of this experiment it helps to look at the data in graphical form. Use a full page of graph paper. Present time along the horizontal axis and temperature along the vertical axis. Plot warming and cooling data on one graph. Also plot the water bath data on the same graph. Draw smooth curves to represent the warming and cooling behavior of the *paradichlorobenzene*. Draw a dotted smooth curve for the water bath data. Be sure to identify each curve.

See Figure 11.

- 2) Write a report summarizing the results of this experiment. Compare the shape of the water bath curves to the shape of the *paradichlorobenzene* warming and cooling curves. Give an explanation to account for any observed differences. Include in the report your interpretation of the shapes of these curves.

Student reports.

- 3) What effect would increasing the amount of *paradichlorobenzene* have on the shape of the curve?

Increasing the amount of substance would not change the fundamental shape of the curve. The melting and freezing temperatures would be constant. The slope of the curve would be more gradual both above and below the plateau providing the same energy source is used. The plateau would be extended.

- 4) From your data, what is the melting temperature for *paradichlorobenzene*?

See 5 for answer.

- 5) From your data, what is the freezing temperature for *paradichlorobenzene*?

The answer to both 4 and 5 is the same: 53°C. Any answer between 51 and 53°C is good considering the uncertainties in the measurements. There is a tendency for the students to obtain higher freezing temperatures than melting temperatures. This is probably due to the poor heat conductivity of solid *paradichlorobenzene*.

A QUESTION TO WONDER ABOUT

Why do the warming and cooling curves have the characteristic shape shown by your graph?

ADDITIONAL INVESTIGATIONS

Determine the cooling behavior of some other pure substance. Your teacher will suggest suitable substances.

There are many organic compounds with relatively low melting temperatures, but most of them tend to undercool excessively. Two that have been tried and have been found to behave well are ethylene carbonate (m.t. = 35–37°C) and benzophenone (m.t. = 47–48°C). Have the students use cooling baths about 15 degrees below the melting temperature.

Experiment 13

Energy Needed to Melt Ice

PURPOSE AND SUMMARY

To determine the amount of thermal energy needed to melt one gram of ice.

The student uses ice to cool a measured amount of water to the ice melting temperature. Assuming no energy loss to the environment he can calculate the energy used to melt the ice using the Δt for the water. The difference between the final volume of warm water plus melted ice and the initial volume of warm water gives the mass of ice melted. The energy transferred from the water as it is cooled can be calculated using the definition of a calorie.

MATERIALS NEEDED

per student or pair

- | | |
|---|--|
| 1 beaker, 250 ml | 1 burner, ring stand, ring, and wire gauze |
| 1 Styrofoam cup, 12 oz | 1 crucible tongs |
| 1 thermometer, -20 to 150°C | 5 or 6 ice cubes. See Lab Hints 1 and 2. |
| 1 graduated cylinder, 100 ml | |

LAB HINTS

1. The ice cubes should be wet, that is, at melting temperature. They should not be used immediately after being removed from the refrigerator if much below the freezing temperature. This is not very likely, since it takes only a few minutes to warm the ice to its melting temperature.
2. Styrofoam ice chests are very efficient in keeping ice throughout the day.

PRELAB DISCUSSION

Little prelab discussion is necessary if the students have done Experiment 12. Most of the students will already know that energy is required to melt ice. Thus, the experiment is simply to determine how much energy. Answer any questions they have concerning the procedure. Remind them to shake excess water from the ice cubes but not to blot them dry. When the ice is removed at the end of the experiment, excess water is drained back into the cup calorimeter.

PRECAUTIONS

Observe the usual laboratory precautions.

POSTLAB DISCUSSION

You might begin discussion by asking the students why they can neglect any temperature change which the water formed by melting ice may have experienced. Ask about the temperature of the water as it comes from melting ice. Water from melting ice is at 0°C . Some

is warmed by mixing and interacting with warmer water, but is later cooled as it interacts with ice. Since the equilibrium temperature is 0°C the energy given up by the water as it cools is equivalent to the energy required to melt the ice. We can neglect any exchange with the environment. There are partially compensating factors involved; warm water is cooled by air at room temperature and cold water is warmed by it. Some ice is also melted by the air.

SAMPLE DATA

volume of warm water used	$100 \pm 1 \text{ ml}$
initial temperature of water	$44.0 \pm 0.2^{\circ}\text{C}$
equilibrium temperature of ice-water system	$0.5 \pm 0.2^{\circ}\text{C}$
volume of water, final	$156 \pm 1 \text{ ml}$

RESULTS OF PROCESSING DATA

- 1) Determine the mass of ice melted. The density of water is one gram per milliliter.

$$\begin{aligned} 156 \text{ ml} - 100 \text{ ml} &= 56 \text{ ml} \\ 56 \text{ ml} \times 1 \text{ g/ml} &= 56 \text{ g} \end{aligned}$$

- 2) Determine the change in temperature of water, Δt .

$$\begin{aligned} \Delta t &= \text{final temperature} - \text{initial temperature} \\ \Delta t &= 0.5 - 44.0 = -43.5 \text{ or } 44^{\circ}\text{C}^{\circ} \text{ (absolute value)} \end{aligned}$$

- 3) Calculate the energy released by 100 g of liquid water as it is cooled through Δt .

$$\begin{aligned} \text{energy given up by 100 g of water} &= \Delta t \times 100 \text{ g} \times 1 \text{ cal/g(C}^{\circ}\text{)} \\ \text{energy} &= 44 \times 100 \times 1 = 4400 \text{ cal} \end{aligned}$$

- 4) Calculate the calories of energy required to melt one gram of ice.

$$\begin{aligned} \text{energy} &= 4400 \text{ cal}/56 \text{ g} \\ &= 79 \text{ cal/g} \end{aligned}$$

- 5) Using the results of this experiment, determine the number of calories required to melt one mole of ice.

$$(79 \text{ cal/g}) \times (18 \text{ g/mole}) = 1400 \text{ cal/mole}$$

- 6) Write an equation for the fusion (melting) of ice including the kcal/mole necessary for melting.



Experiment 14

Energy of Crystallization

PURPOSE AND SUMMARY

To observe the sudden rise in temperature which occurs as undercooled sodium thiosulfate pentahydrate crystallizes, and to give the student the opportunity to plan an experiment on his own. To calculate the energy released as one gram of the substance is crystallized.

The student prepares some undercooled sodium thiosulfate, and induces rapid crystallization by *seeding*. He observes the sudden rise in temperature as the energy of crystallization is released. Then he is given the opportunity to plan his own experiment and to make the necessary measurements and observations in order to calculate the energy of crystallization as calories per gram.

TIMING

After Experiment 13 and before Section 5-2 in the Textbook.

It will take from 20 to 30 minutes for the qualitative part of the experiment. Once the quantitative experiment has been planned it can be done in about 30 minutes.

MATERIALS NEEDED

per student or pair

- 1 test tube, 18 × 150 mm
- 2 thermometers, -20° to 150°C
- 1 test tube clamp
- 1 burner
- 1 Styrofoam cup, 12 oz
- 1 graduated cylinder, 100 ml
- 1 balance, decigram
- 20 grams sodium thiosulfate pentahydrate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5 \text{H}_2\text{O}$)

LAB HINT

Have reagent bottles of sodium thiosulfate near each balance. You may wish to have the students weigh the sodium thiosulfate on weighing papers and then transfer the crystals to the test tube.

PRELAB DISCUSSION

The prelab discussion could be centered around the questions brought up in the introduction to the experiment. Let the students talk and express their ideas, but do not give away the discovery aspect of the experiment.

PRECAUTIONS

Tell the students to be careful not to overheat the substance above 70 or 80°C. After it is all melted the temperature rises rapidly.

POSTLAB DISCUSSION

It is important that students have an opportunity to plan experiments on their own. Some will be able to do this much better than others. Even though it may be extremely difficult for some, the effort put forth in trying to think through a problem and having a chance to try their own experiment can be very rewarding. After the students have completed the first part of the experiment, give them a day or two to work on the problem. Those who are able to invent a procedure should either be given the opportunity to go ahead with their experiment or present their plan to the class for discussion.

During the class discussion, a procedure should be outlined by the students. The plan should include how they intend to treat the data after they get it. Another approach is to let those who are able, proceed with their own experiment, and give a procedural outline to the students who are unable to plan on their own. See *Suggested Procedure and Processing the Data*.

QUESTIONS TO BE CONSIDERED WHILE PLANNING THE EXPERIMENT WITH A CLASS OF STUDENTS

- 1) What can we use to absorb the energy released as the sodium thiosulfate solidifies? A Styrofoam plastic cup containing cold water makes a good calorimeter.
- 2) How much water should be used? The smaller the amount of water the larger the Δt . Use enough so that the level of sodium thiosulfate in the test tube will be just below the level of the water in the cup.
- 3) What should the water temperature be at the time the sodium thiosulfate is seeded? It should be low enough so that the sodium thiosulfate can cool again to its starting temperature after solidification is complete. The water should be about 10 to 15 degrees cooler than the sodium thiosulfate at the time they are put together. The cold water in the calorimeter will warm a little and the sodium thiosulfate will cool when they are placed together before seeding. However, it makes no difference to the outcome of the experiment whether the seeding is done immediately or several minutes later. The increase in temperature of the the water in the calorimeter can only be from two sources; interaction with air and energy released during solidification of the sodium thiosulfate.

Cold water will warm a little due to energy transfer from the air. You may want to set up a control system unless this effect is to be neglected. 100 ml of water at 15°C contained in a Styrofoam cup will warm only about 0.5°C per 10 minutes due to air interaction.

- 4) Is it necessary to keep track of the temperature of the sodium thiosulfate during solidification? It isn't really necessary to obtain time-temperature data, however, it is interesting and very helpful to be able to see the relation between the two curves of the graphed data. It is also easier to determine Δt correctly using graphed data. Delta t is the change in temperature of the water as the sodium thiosulfate temperature changes and returns to the starting temperature again.

SUGGESTED PROCEDURE

- f. Melt and cool the weighed sodium thiosulfate as in steps a, b, and c of the earlier procedure.
- g. Prepare a cold water bath in a Styrofoam cup. Use 100 ± 1 ml of water between 10 and 15°C .
- h. Record the temperature of both the sodium thiosulfate and the water bath just before putting them together. Place the test tube containing liquid sodium thiosulfate in the cold water bath, then drop a small crystal of sodium thiosulfate into the test tube. Obtain time-temperature data at one minute intervals until the temperature of the sodium thiosulfate returns to the same temperature it had when placed into the calorimeter.
- i. Plot the time-temperature data for both the sodium thiosulfate and the water on the same graph.

PROCESSING THE DATA

- 1) Determine the Δt for the water in the calorimeter. Δt is the change in water temperature as the sodium thiosulfate temperature changes and returns to the starting temperature again.
- 2) Calculate the calories used to warm the water through Δt .
- 3) Calculate the calories per gram given up by the sodium thiosulfate as it solidifies.
- 4) Calculate the percent error in your cal/gram determination. Your teacher will give you the accepted value from the literature.
- 5) From your data, what is the freezing temperature of sodium thiosulfate pentahydrate?

SAMPLE DATA

Mass of Sodium Thiosulfate 20.0 g

Time-Temperature Data

Time, min.	Temperature, $^{\circ}\text{C}$	
	Water Bath	Sodium Thiosulfate
0	14.5	32.0
1	17.5	47.5
2	19.5	48.2
3	21.5	48.2
4	22.0	48.2
5	23.0	46.5
6	23.3	44.0
7	23.5	40.0
8	23.7	32.0

RESULTS OF PROCESSING THE DATA

- 1) Determine the Δt for the water in the calorimeter. Δt is the change in the temperature of the water as the sodium thiosulfate temperature changes and returns to the starting temperature again.

$$\begin{aligned}\Delta t &= \text{final temperature} - \text{initial temperature} \\ &= 23.7 - 14.5 = 9.2^\circ\text{C}\end{aligned}$$

- 2) Calculate the calories used to warm the water through Δt temperature change.

$$\begin{aligned}\text{calories to warm water} &= \text{mass of water} \times \Delta t \times 1 \text{ cal/g}(^\circ\text{C}) \\ &= 100 \text{ g} \times 9.2^\circ\text{C} \times 1 \text{ cal/g}(^\circ\text{C}) \\ &= 920 \text{ cal}\end{aligned}$$

- 3) Calculate the calories per gram given up by sodium thiosulfate as it solidifies.

$$\text{calories per gram sodium thiosulfate} = 920 \text{ cal}/20.0 \text{ g} = 46 \text{ cal/g}$$

The accepted value from the literature is 47.8 cal/g.

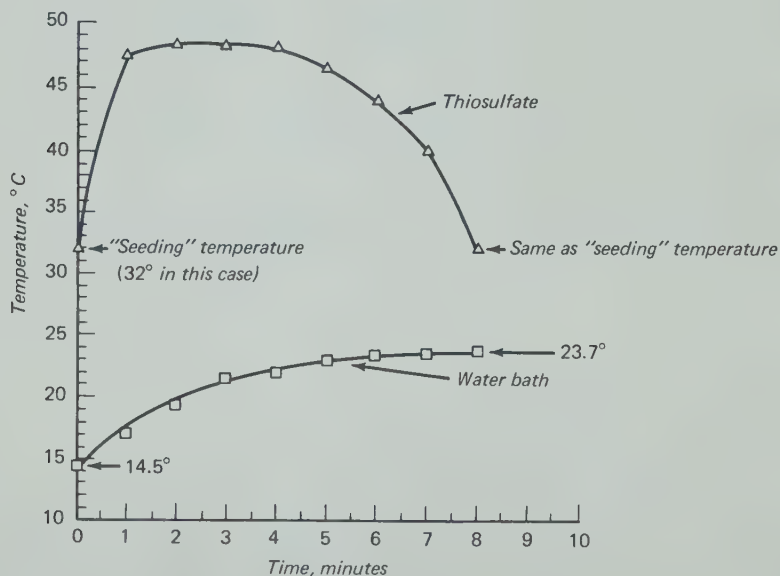


Figure 12 Time-temperature data for $\text{Na}_2\text{S}_2\text{O}_3$ crystallization.

- 4) Calculate the percent error in your cal/gram determination.

$$\begin{aligned}\text{percent error} &= \frac{(\text{accepted value} - \text{experimental})}{\text{accepted value}} \times 100 \\ &= \frac{47.8 - 46}{47.8} \times 100 \quad \text{or} \quad \frac{48 - 46}{48} \times 100 = 4.2\%\end{aligned}$$

- 5) From your data, what is the freezing temperature of sodium thiosulfate pentahydrate? The flat portion of the curve indicates the freezing temperature, 48.2°C. The accepted value from the literature is 48.0°C.

DEMONSTRATION 16
EQUIVALENCE OF LIQUID AND VAPOR TEMPERATURE
FOR A BOILING SAMPLE

PURPOSE

To show that the temperature of a vapor above a boiling liquid is not greater than the temperature of the liquid.

TIMING

During Section 5-3. Perhaps 10 minutes required.

MATERIALS NEEDED

- 1 Erlenmeyer flask, 500 ml
- 2 thermometers, -20 to 150°C
- 2 corks to hold thermometer
- 1 ring stand, ring, wire gauze
- 2 clamps
- 1 lab burner

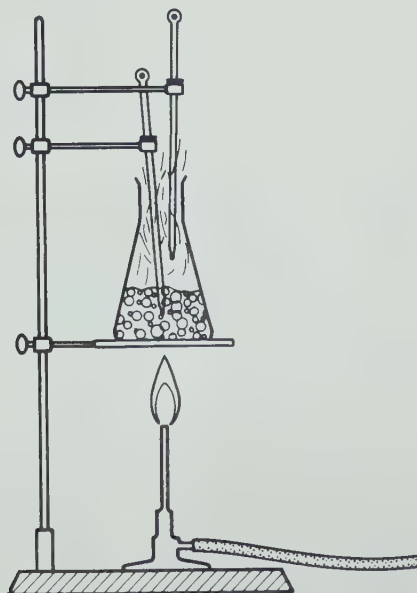


Figure 13
Measuring the temperature
of a boiling liquid and its vapor.

CLASSROOM PRESENTATION

Set up the apparatus as in Figure 13. Have a student read and record the temperatures every few minutes as the liquid is being heated. The temperature of the vapor will probably not get as high as that of the liquid. Do not use a large flame which might superheat the vapor.

FOLLOW-UP

This demonstration is needed to convince students that the temperature of steam is not greater than that of boiling water. To accept this seems to go against the intuition of many students. This simple example will be convincing proof. Emphasize that the observed behavior is general and that, when carefully done, the liquid and vapor temperatures are equal.

DEMONSTRATION 17

VAPOR PRESSURE OF A LIQUID AT TWO TEMPERATURES

PURPOSE

To show that liquids have a vapor pressure; to show that this pressure rises as temperature increases.

TIMING

This demonstration should be given after the student has read Section 5-4. About 20 minutes are needed.

MATERIALS NEEDED

apparatus described in the Lab Hint
1 to 3 large Erlenmeyer or Florence flasks, 250 or 500 ml
3 hole rubber stopper to fit flask
dropping funnel
mercury manometer, 55–60 cm
pinch clamp
glass tubing, two 90° bends plus tubing for manometer
rubber tubing

LAB HINT

The apparatus may be constructed as shown in Figure 14.

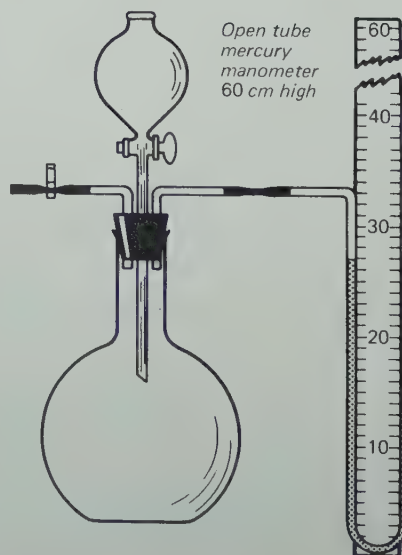


Figure 14
Vapor pressure apparatus.

CLASSROOM PRESENTATION

Partially evacuate a flask. A pressure of about 600 mm (160 mm difference in manometer levels) is convenient. Evacuate the flask through the tube with the pinch clamp. Close the pinch clamp, and watch the manometer. If it shows no change in 2–3 minutes, there are no important leaks. Record the initial pressure by reading the level in each arm.

Place a few milliliters of acetone in the dropping funnel. Open the stopcock to allow a few drops to enter the flask. The movement of the mercury levels will be immediately visible. Record levels every 2–3 minutes until no more change is evident (about 15 minutes). Introduce another few drops of acetone to be sure all that will evaporate at this temperature has done so.

By this procedure you should observe a vapor pressure of about 150 mm. Some cooling will take place during evaporation, hence the value will be for a temperature probably less than 20°C. Repeat the procedure with the flask immersed in a warm water bath, or have two sets of apparatus and run the two trials simultaneously.

Vapor pressures for acetone are :

Temp. (°C)	Pressure (mm)
15	147
18	169
21	193
24	220

Data from a trial run :

	Open Tube	Tube to Flask	Pressure in Flask
Initial reading	165 mm	345 mm	180 mm
Reading after entire procedure above (20 min)	242 mm	267 mm	25 mm
Change in pressure :			
180 – 25 = 155 mm			

Other calculations are done in the same way.

DISCUSSION

These are the points to make with this experiment.

- 1) Liquids vaporize, giving a definite vapor pressure.
- 2) Vapor pressure goes up as temperature increases. As a secondary point, make it clear that vapor pressure is not changed by adding more liquid.

DEMONSTRATION 18

PREPARATION AND VAPOR PRESSURE OF IODINE

PURPOSE

To show the high vapor pressure of $I_2(s)$.

TIMING

During Section 5-6, 10 minutes are needed.

MATERIALS NEEDED

H_2SO_4 , conc., 10 ml
 H_2O , 5 ml
 $\text{MnO}_2(\text{s})$, 5 grams
 $\text{NaI}(\text{s})$, 5 grams
beaker, 250 ml
evaporating dish, slightly larger than top of beaker
ice
ring stand, ring, wire gauze
lab burner

PROCEDURE

Carefully add the conc. H_2SO_4 to water and MnO_2 in the beaker. With care, add the NaI . Place evaporating dish containing ice on top of beaker to act as condenser. When good platelets of $\text{I}_2(\text{s})$ have collected on the bottom of the evaporating dish remove it from the beaker. You may have to heat the beaker to speed reaction and drive off the I_2 vapor.

FOLLOW-UP

Call attention to the crystals and put them on display in a well ventilated place. I_2 vapors irritate mucous membranes. Next day, call attention to their absence. They will have sublimed. The vapor pressure of $\text{I}_2(\text{s})$ at 25°C is about 0.3 mm of Hg.

Solutions, Solubility, and Ions



After studying this chapter the student should have a good start toward a knowledge of solutions. In particular he should know :

- 1) What is meant by solubility and that it varies for different compounds.
- 2) Some solutions conduct electricity and hence are deduced to contain ions.
- 3) Behavior of electric charges are important to chemistry and that like charges repel while unlike ones attract each other.

The direct experimental work of the student deals with solubility. There are several demonstrations and a film to provide the information about conductivity.

Schedule of Experimental Work

TEXT SECTION	EXPERIMENTAL WORK	FOR DETAILS SEE PAGE
6-1	Expt. 15	82
	Expt. 16	87
6-2, 6-3	Demo. 19	92
	Demo. 20	92
6-4 to 6-7	Demo. 21	93
	Expt. 17	95
6-8, 6-9	Demo. 22	99
	Demo. 23	100
6-10	Expt. 18	101
6-11		

Experiment 15

Cooling Behavior of a Solution

PURPOSE AND SUMMARY

To observe the cooling behavior of a solution as compared to the cooling behavior of a pure substance. To make a quantitative study of the melting temperature of a pure substance when measured amounts of solute are added to it.

The student first observes the cooling behavior of acetamide in order to determine its melting temperature. He then adds 0.0025 mole of a solute to the acetamide and again observes the cooling behavior to determine the temperature at which the solution begins to freeze. An additional 0.0025 mole of the same solute is added and the freezing temperature is determined. Using class data the students are able to search for regularities which may be apparent from the data.

TIMING

Before Section 6-1 in the Textbook.

One full period will be needed for most students. Some may finish in time to do the suggested additional investigation.

MATERIALS NEEDED

per student or pair

1 test tube, 18 × 150 mm

5 grams of acetamide, reagent grade

1 test tube clamp

1 burner, ring stand, and clamp

1 balance, centigram

1 thermometer, -20° to 150°C

0.0050 mole of: urea, NH_2CONH_2 ; camphor, $\text{C}_{10}\text{H}_{18}\text{O}$; sodium iodide, NaI ; or potassium iodide, KI

per class

clock with sweep-second hand

LAB HINTS

- 1) If bulk pan balances are available, have the students weigh the 5.0 grams of acetamide using these balances with weighing papers. Use centigram balances, however, to weigh the solutes.
- 2) Sodium iodide is quite hygroscopic. It should be dried in an oven and kept dry in a dessicator when not being used to weigh out samples.

PRELAB DISCUSSION

Review the procedure briefly. Give instructions on how to weigh the 5.0 gram sample of acetamide, and the very small amounts of solutes. The uncertainty in mass of solutes will range between about 5 and 13% when weighing with a weighing paper on a centigram balance. The following samples contain 0.0025 mole.

urea	0.15 ± 0.02 g
camphor	0.38 ± 0.02 g
sodium iodide	0.40 ± 0.02 g
potassium iodide	0.42 ± 0.02 g

You may wish to reduce this uncertainty by doubling the amount of acetamide and the amount of solute used in the experiment.

Remind the students that all of the solute must be dissolved before they begin taking time-temperature cooling data. In step b the student is directed to stir with a thermometer until a constant temperature has been reached during solidification. It is not necessary to continue until all the acetamide has solidified. They should continue just long enough to determine the freezing temperature.

Excessive undercooling can be avoided by occasionally rubbing the bulb of the thermometer along the inside of the test tube to pick up a few crystals and then returning it to the liquid. The tiny crystals act as nuclei on which larger crystals can begin to form. If extreme undercooling occurs, suggest that they repeat the experiment using the same solution warming it just above the freezing temperature and then repeating as before.

PRECAUTIONS

Observe usual laboratory precautions. Tell the students not to overheat the acetamide or the solutions. Temperatures over 100°C should be avoided.

POSTLAB DISCUSSION

Students may need help with extrapolation of the freezing curves in order to determine the temperature at which the solutions begin to freeze.

Using class data, help the students search for regularities. Within the limits of uncertainty they might conclude that 0.0025 mole of KI or NaI is about as effective in lowering freezing temperature of acetamide as 0.0050 mole of either camphor or urea. Since doubling the number of moles of solute approximately doubles the Δt_f , they might conclude that the number of particles present determines Δt_f . The two iodides ionize to produce 2x moles of ions for each x moles of solid dissolved.

Discuss ways of improving the precision of the experimental data.

SAMPLE DATA

freezing temperature for acetamide = 79.7°C

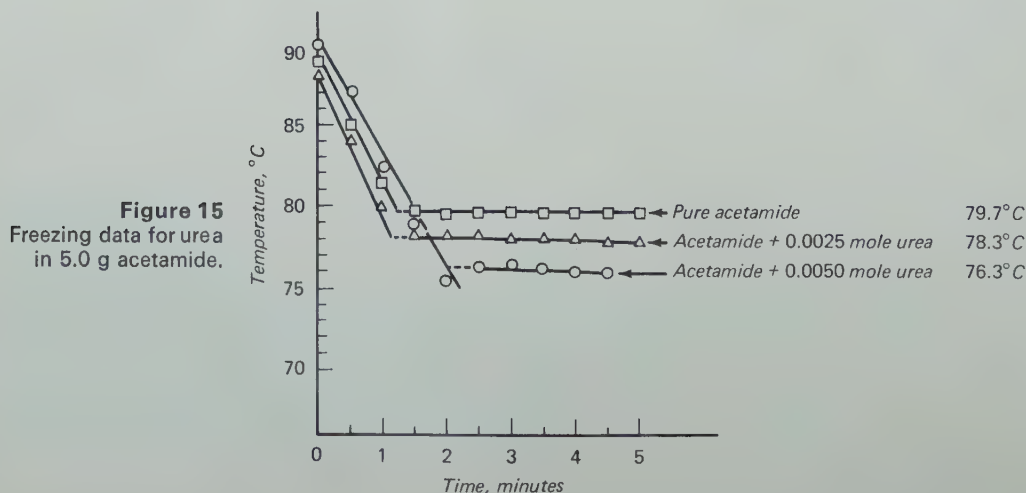
Solution of acetamide plus 0.0025 mole of solute	freezing temperature	Δt_f
urea	78.3	1.4
camphor	78.0	1.7
Nal	75.0	4.7
KI	75.3	4.4

Solution of acetamide plus 0.0050 mole of solute	freezing temperature	Δt_f
urea	76.3	3.4
camphor	76.5	3.2
Nal	69.5	10.2
KI	70.8	8.9

RESULTS OF PROCESSING THE DATA

- 1) Plot, on the same graph, the time and temperature data from all three determinations. Present temperature along the vertical axis and time along the horizontal axis. Choose appropriate scales. Label each curve. For the solutions, indicate how much of the assigned solute was dissolved in acetamide. Sometimes these solutions tend to under-cool. That is, they cool to a temperature below the freezing temperature before crystal formation begins. When crystallization begins, the temperature suddenly rises. This sudden change in temperature is due to the release of thermal energy. The freezing temperature is obtained by extrapolation as shown in Figure 15-2 in the Lab Manual.

Figures 15 to 19 show typical data.



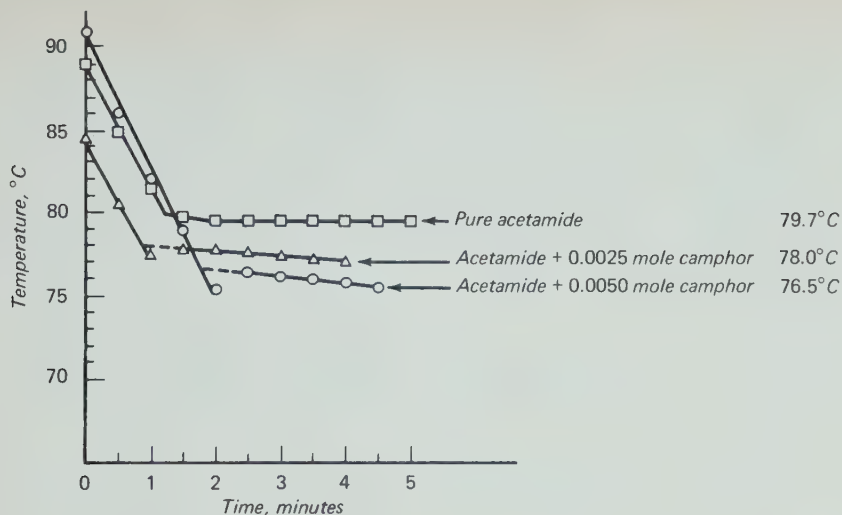


Figure 16
Freezing data for camphor
in 5.0 g acetamide.

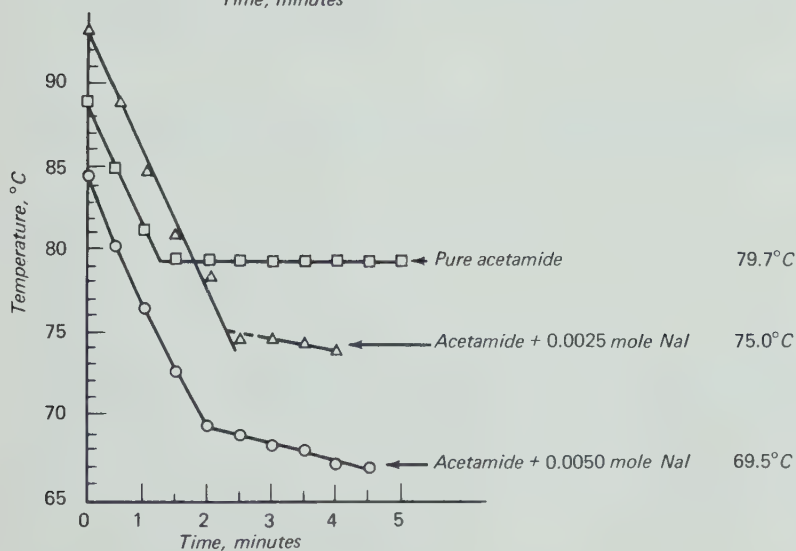


Figure 17
Freezing data for NaI
in 5.0 g acetamide.

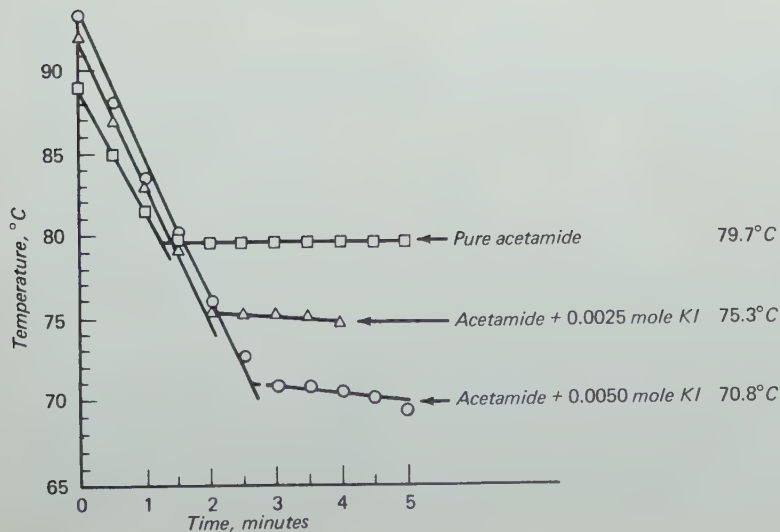
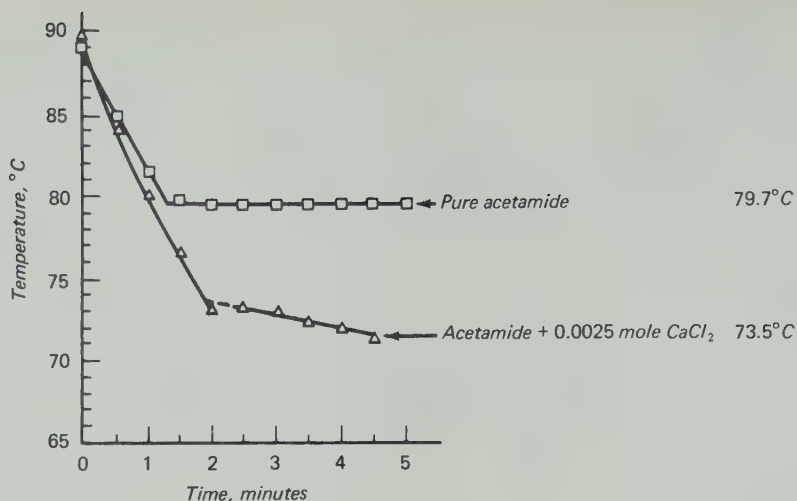


Figure 18
Freezing data for KI
in 5.0 g acetamide.

Figure 19
Freezing data for CaCl_2
in 5.0 g acetamide.



- 2) Determine the change in freezing temperature, Δt_f , caused by adding 0.0025 mole of solute to acetamide.

urea	1.4 C°
camphor	1.7
Nal	4.7
KI	4.4

- 3) Determine the additional change in freezing temperature, Δt_f , when the second amount of solute was added.

urea	2.0 C°
camphor	1.5
Nal	5.5
KI	4.5

- 4) Average the Δt_f values for each 0.0025 mole of solute added.

urea	1.7 C°
camphor	1.6
Nal	5.0
KI	4.4

- 5) Compare average values of Δt_f when 0.0025 mole of each solute was added to 5.0 grams of acetamide. Search for a regularity among these data.

Class data should show that Nal and KI both lower the freezing temperature about twice as much as do either urea or camphor. Urea and camphor dissolve in acetamide to produce molecules in solution, while Nal and KI dissolve to form ions in solution. A mole of either Nal or KI will produce 2 moles of particles in solution, while a mole of either urea or camphor will produce just one mole of particles in solution.

- 6) The temperature remained essentially constant during the solidification of acetamide. During the solidification of the solutions the temperature dropped. Explain.

As the acetamide freezes out of solution, the remaining solution becomes more and more concentrated. Thus, the freezing temperature continues to get lower and lower as the solution becomes more concentrated.

ADDITIONAL INVESTIGATION

Predict how much the freezing temperature for acetamide will be lowered by the addition of 0.0025 mole of anhydrous CaCl_2 . After making your prediction do an experiment to check it out.

One mole of CaCl_2 dissolved in solution should produce three moles of ions, thus the freezing temperature of 5.0 grams of acetamide should be lowered about three times as much as that for the molecular solids and about three-halves as much as the ionic solids.

Experiment 16

Formula of a Precipitate

PURPOSE AND SUMMARY

By providing an excess of one and then an excess of another chemical, the student can see that only a certain amount of one chemical (Pb^{2+}) will combine with a certain amount of another (I^-). He also gains experience in preparing and manipulating solutions.

Students work in pairs. Each prepares a 0.50 *M* solution. One of $\text{Pb}(\text{NO}_3)_2$ the other of NaI . These are combined in several ratios. The moles of Pb^{2+} and of I^- present in the combinations that gave the greatest amount of precipitate are used to determine its chemical formula.

TIMING

After Section 6-2. About 50 minutes to finish through step i on the first day. Another 20 minutes are needed the next day to filter the solutions. A few minutes are needed to weigh the dry precipitates on the third day.

MATERIALS NEEDED

per student

- 1 beaker, 100 ml
- 1 graduated cylinder, 50 ml
- 1 buret assembly, Mohr, 25 ml. See Lab Hint 2.
- 4 test tubes, 13 × 100 mm. See Lab Hint 3.
- 1 funnel, 65 mm and holder
- 1 filter paper disk, 11 cm. See Lab Hint 4.
- thumb protectors. See Lab Hint 5.

per class

balances, centigram
bottles of $\text{Pb}(\text{NO}_3)_2(\text{s})$, one per four students

per pair

vials of $\text{NaI}(\text{s})$. See Lab Hint 1.
distilled water, 1 squeeze bottle (300 ml)
buret clamp, double
ring stand
Bunsen burner
ring, 4" and wire gauze
drying apparatus. Either an oven or heat lamps are suitable.

LAB HINTS

- 1) NaI is deliquescent. It is best dispensed by issuing a vial containing about 4 g (3.74 g required) to each student needing it. Use of KI is discouraged because 332 g of KI react with 331 g $\text{Pb}(\text{NO}_3)_2$. This coincidence could mislead the student into thinking one gram of one substance reacts with one gram of another. NaI is better because 300 g are needed. Have labeled jars available for waste $\text{NaI}(\text{s})$, and others for waste $\text{Pb}(\text{NO}_3)_2(\text{s})$.
- 2) The glass bead-rubber tube buret valve is ingenious, simple, safe, and economical. Explain that its operation depends on proper squeezing to make a space between the glass bead and rubber wall.
- 3) Tubes can be numbered with
 - wax pencil
 - bits of masking tape
 - prepared labels (Be sure the adhesive sticks to glass.)
- 4) Rather than have each student weigh a piece of filter paper, reduce the percent error by having one student weigh 100 pieces. Post the mass of one piece.
- 5) Small plastic squares (2") cut out of plastic bags from dry cleaned garments make excellent substitutes for corks. They can be discarded after one use. To make, fold plastic into strips about $2\frac{1}{2}$ " wide. With scissors cut from this 2" strips with folds along the 2" edges. Cut off and discard the folds. This procedure produces packs of squares from which single squares can be peeled. The student holds the plastic over the test tube, shakes the solution, and discards the plastic.

PRELAB DISCUSSION

The student should clearly understand that the main object of this experiment is to determine the formula of a precipitate. Another is to answer the question, "Does the amount of precipitate formed depend on the amount of Pb^{2+} , the amount of I^- , or both?"

Emphasize the observation of significant figures. For example 50.0 ml means 50.0 ± 0.1 ml, while 5 ml means 5 ± 1 ml.

Explain the danger of returning a chemical to a stock bottle, Step b. Trying to save a little is likely to waste a lot.

Pose the problem of identifying an unlabeled solution in beaker or buret. Simply test it against a solution known to contain either Pb^{2+} or I^- .

The reason for mixing solutions as in step h is to produce large crystals. Very small crystals will pass through or clog filter paper.

Cold water is needed in step l to minimize dissolving the precipitate. Solubility of PbI_2 in 100 ml water at 0° , 20° , and 100° is 0.044, 0.063, and 0.41 g.

Disposing of the filtrate in step n can create a fair problem solving situation. Have the students combine filtrates within a group. A precipitate will form. Why? Some filtrates contain Pb^{2+} , and others I^- ions.

A second drying and weighing of precipitates in step o is recommended if time allows.

It pays to check that each student has calculated the correct amount of solid needed to make the solutions.

$$0.50 M = \frac{0.50 \text{ mole}}{\text{liter of sol'n}} = \frac{0.0250 \text{ mole}}{50.0 \text{ ml of sol'n}}$$

1 mole NaI weighs 150 g

$$\frac{150 \text{ g}}{\text{mole}} \times \frac{0.0250 \text{ mole}}{50.0 \text{ ml of sol'n}} = \frac{3.74 \text{ g NaI}}{50.0 \text{ ml } 0.50 M \text{ sol'n}}$$

1 mole $\text{Pb}(\text{NO}_3)_2$ weighs 331 g

$$\frac{331 \text{ g}}{\text{mole}} \times \frac{0.0250 \text{ mole}}{50.0 \text{ ml of sol'n}} = \frac{8.28 \text{ g } \text{Pb}(\text{NO}_3)_2}{50.0 \text{ ml } 0.50 M \text{ sol'n}}$$

PRECAUTIONS

Observe the usual lab precautions. Sloppy technique is readily revealed by the appearance of a premature yellow precipitate.

POSTLAB DISCUSSIONS

This is best handled by going over the student write-ups.

PROCESSING THE DATA AND SAMPLE DATA

- 1) Obtain the mass of the dried precipitate from each member of your group. From this, determine the average grams of lead iodide in each tube. Be sure the mass of the filter paper is not included.

See Table 16-1 for a complete tabulation of calculations.

- 2) Determine the number of millimoles of $\text{Pb}(\text{NO}_3)_2$ and of NaI added to each tube.

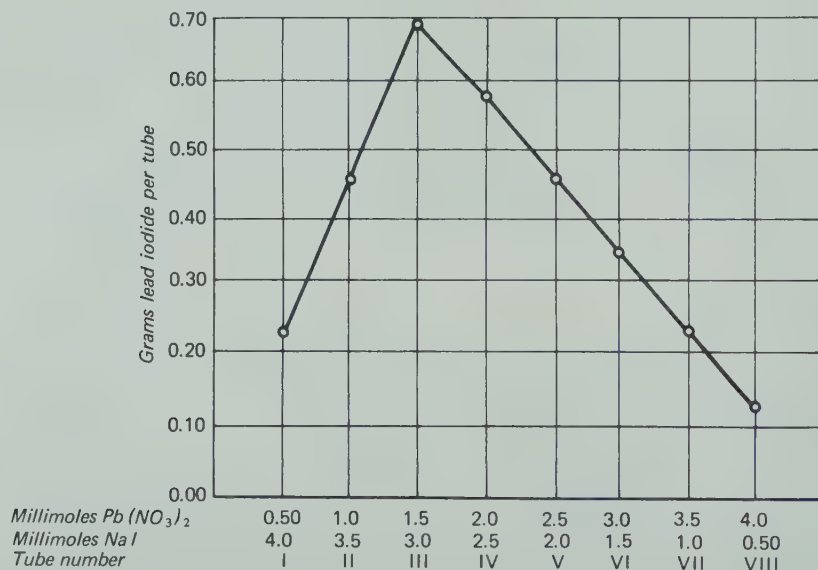
Since each solution is $0.50 M$, each ml will contain 0.00050 mole of solute or 0.50 millimole.

Tube No.	I	II	III	IV	V	VI	VII	VIII
ml $\text{Pb}(\text{NO}_3)_2$ sol'n	1	2	3	4	5	6	7	8
millimoles Pb^{2+}	0.50	1.0	1.5	2.0	2.5	3.0	3.5	4.0
ml NaI sol'n	8	7	6	5	4	3	2	1
millimoles I^-	4.0	3.5	3.0	2.5	2.0	1.5	1.0	0.50

- 3) Present your results in graphical form. Show millimoles of $\text{Pb}(\text{NO}_3)_2$ along the horizontal axis starting at the origin. Show millimoles of NaI along the same horizontal axis but finishing at the origin. As a third label for this axis, show tube number. See Figure 16-4. Show grams of lead iodide along the vertical axis. Label the graph and each axis.

See Figure 20.

Figure 20
Grams of lead iodide formed.



- 4) How many millimoles of Pb^{2+} were available when the greatest amount of lead iodide was formed?

1.5 millimoles of Pb^{2+} were available when the greatest amount of lead iodide formed.

- 5) How many millimoles of I^- were available when the greatest amount of lead iodide was formed?

3.0 millimoles of I^- were available when the greatest amount of lead iodide formed.

- 6) What is the simplest whole number ratio of $\frac{\text{moles } \text{I}^-}{\text{moles } \text{Pb}^{2+}}$ when the greatest amount of lead iodide was formed?

When the greatest amount of lead iodide formed, the ratio was

$$\frac{\text{moles I}^-}{\text{moles Pb}^{2+}} = \frac{3.0}{1.5} = \frac{2}{1}$$

7) What is the formula of lead iodide?

The formula of lead iodide is PbI_2 .

8) Why was the same amount of lead iodide not formed in each tube?

To form PbI_2 , one mole of Pb^{2+} requires 2 moles of I^- . This ratio of amounts was only present in tube III. The solutions in the other tubes were deficient in one ion.

9) According to this experiment, when the amount of lead iodide formed was a maximum, how many moles of lead iodide were formed for each 1.00 mole of $\text{Pb}(\text{NO}_3)_2$ used?

In tube III there was

$$\frac{0.69 \text{ g PbI}_2}{461 \text{ g PbI}_2/\text{mole}} = 0.0015 \text{ mole PbI}_2$$

There was 0.0015 mole $\text{Pb}(\text{NO}_3)_2$ in tube III. The ratio $\frac{\text{moles PbI}_2}{\text{moles Pb}(\text{NO}_3)_2} = \frac{1}{1}$. This

assumes all the Pb^{2+} in tube III was actually used. Further experimentation as a class demonstration could test this assumption.

10) Write a balanced equation for the formation of lead iodide from ions.

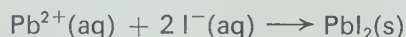


TABLE 16-1

Tube Number	I	II	III	IV	V	VI	VII	VIII
ml 0.50 M $\text{Pb}(\text{NO}_3)_2$	1	2	3	4	5	6	7	8
millimoles Pb^{2+}	0.50	1.0	1.5	2.0	2.5	3.0	3.5	4.0
ml 0.50 M NaI	8	7	6	5	4	3	2	1
millimoles I^-	4.0	3.5	3.0	2.5	2.0	1.5	1.0	0.50
millimoles PbI_2	0.50	1.0	1.5	1.25	1.0	0.75	0.50	0.25
g PbI_2 (class results)	0.23 (0.23)	0.46 (0.46)	0.69 (0.68)	0.58 (0.58)	0.46 (0.46)	0.35 (0.38)	0.23 (0.27)	0.12 (0.14)
g $\text{PbI}_2 \times 4$	0.92	1.84	2.76	2.32	1.84	1.40	0.92	0.48

DEMONSTRATION 19**MODELS TO SHOW SOME RELATIVE SOLUBILITIES**

PURPOSE

To show relative solubilities of some common substances.

TIMING

A few minutes are needed during Section 6-3.

MATERIALS NEEDED

The amount of solid that will dissolve to make 1 liter of saturated solution is represented by a cube of appropriate volume.

Cubes can be made of cardboard as described in Demonstration 8, p. 30, or of wood. Small cubes can be cut from solid wood. Larger cubes are best made by cutting six squares, with edges beveled at 45°, from 1/4" plywood. Glue and hold in place with rubber bands. The dimension given provides a cube which represents the volume of solid that will dissolve in 1 liter of water at 20°C.

Substance	Length of cube edge, cm	Solubility, g/liter
Reference	10	—
Sucrose	10.9	2040
$\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$	5.2	325
NaCl	5.5	360
PbCl_2	1.2	9.9

Paint each cube an appropriate color, and label one face.

CLASSROOM PRESENTATION

Simply show the cubes stating what they represent. Discussion of factors influencing solubility is best postponed until Chapter 13.

DEMONSTRATION 20**SOLUBILITY VS TEMPERATURE**

PURPOSE

To show a change of solubility with temperature in a very interesting fashion.

TIMING

A few minutes are needed early in a period during Section 6-3.

MATERIALS NEEDED

some $\text{Pb}(\text{NO}_3)_2$ solution
some NaI solution
1 flask, 1 liter
1 cork for flask

PROCEDURE

Concentration and amount of solutions is not critical. Enough is needed to make about 800 ml of saturated solution of PbI_2 . Mix 400 ml 0.01 M $\text{Pb}(\text{NO}_3)_2$ with 400 ml 0.01 M NaI . Filter and save some precipitate.

Heat almost to boiling. Add precipitate until it just disappears as the solution reaches about 90°C .

As the solution cools, beautiful golden platelets of PbI_2 precipitate are produced. Swirling the flask produces a beautiful display of iridescent color. Leave the flask on the demonstration table for a few days.

DEMONSTRATION 21**ELECTRICAL CONDUCTIVITY**

PURPOSE

To demonstrate the electrical conductivity of solutions, and that conductivity depends on concentration.

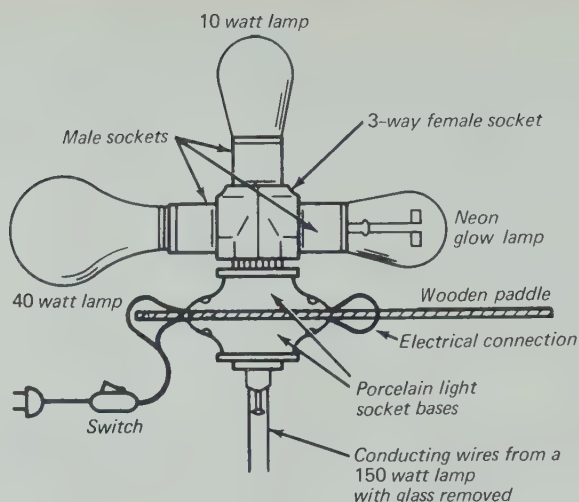
TIMING

About 20 minutes during Section 6-7.

MATERIALS NEEDED

tap water
distilled water
about 50 ml of 1 M solutions of each of the following: NaCl , sucrose, CaCl_2 , Na_2SO_4 ,
 NH_4Cl , $(\text{NH}_4)_2\text{CO}_3$
about 50 ml of 0.1 M solutions of NaCl , AgNO_3 , NaHCO_3 and CuCl_2
about 50 ml of 0.01 M NaCl
15 beakers, 50 ml. See Lab Hint 1.
electrical conductivity apparatus as shown in Figure 21.

Figure 21
Conductivity apparatus.



LAB HINTS

1. The various reagents and materials can be placed in 2-ounce wide-mouth bottles with screw caps, and can be tested directly in the bottles. Most reagents can be stored in these same bottles for use again next year.

PROCEDURE

Have the students record the results in their lab notebooks and, after the experiment, write a summary report accounting for the differences in conductivity that they observe.

Each solution is mentioned in Section 6-7. Show the student that the statements are backed by experiment.

To demonstrate the effect of concentration on conductivity, substitute a 100 or 200 W bulb for the neon glow-lamp. If the probes are bent to about 1 inch apart and immersed to a depth of about 1 inch, a 0.001 *M* solution of NaCl will not cause the 10 W bulb to glow, but a 0.002 *M* solution will. A simple way of showing this is to add 0.1 *M* NaCl to a 50 ml beaker half-full of distilled water. Five small drops (about $\frac{1}{4}$ ml) will produce a 0.001 *M* solution; and 10 drops, about 0.002 *M*. At a concentration of about 0.005 *M*, the 10 W bulb is about at its maximum brightness, and the 40–50 W bulb shows a slight glow. A 50 W bulb does not reach maximum brightness until the concentration is about 0.01 *M*, at which concentration there is no glow in a 200 W bulb. A faint glow appears in the 200 W bulb at about 0.02 *M*. Full brightness is achieved in 0.1 *M* NaCl. This demonstration gives clear evidence that the more ions there are, the greater is the conductivity.

Avoid trying to explain how the ions carry electric current. This is done much better in Chapter 15, Oxidation-Reduction Reactions.

This apparatus permits testing a wide range of conductivities. Two porcelain receptacles are mounted back to back on a piece of plywood (shaped like a Ping-Pong paddle) to permit the apparatus to be mounted on a ring stand or held by hand. The conducting wires are those which held the filament in a broken 150–250 W lamp. The wiring is done as shown in the diagram.

PRECAUTIONS

To avoid an electric shock, always turn off the switch or pull the plug while rinsing the electrodes or changing the solutions.

If only the neon glow-lamp is used (with other lamps loosened in their sockets), it will pick up very weak electrolytes, such as water, 95% alcohol, and glacial acetic acid. The 10 W lamp requires more current, and, as a result, will show the effect of dilution on acetic acid or ammonia. Other, higher wattage lamps can be used to pick up stronger electrolytes. Conductometric titrations, such as $0.01\text{ M H}_2\text{SO}_4 + 0.01\text{ M Ba(OH)}_2$, can be carried out, in which the lamps will be extinguished one after another as the endpoint is reached and then be relighted after it is passed.

Experiment 17

Reactions between Ions in Aqueous Solutions

PURPOSE AND SUMMARY

To introduce the idea of reacting species in a chemical reaction as contrasted with those that are merely *spectators*, and to give the student practice in writing *net ionic equations*. To provide additional experience with differences in solubility. To offer a chance to apply logical thinking processes in order to deduce which ions are responsible for observed precipitates. To show that an ion is a chemical entity with properties independent of its source.

The student mixes six different solutions two at a time until all possible combinations have been tried. From the ions present, he is able to deduce which of the possible ion combinations are responsible for the precipitates.

TIMING

After Section 6-7 in the Textbook.

About 10 minutes is sufficient time to try all the possible combinations using one set of solutions. Additional time is needed to analyze the observations and test other sets of solutions.

MATERIALS NEEDED

per group of four students

1 set of six solutions in dropper bottles. See Lab Hints 1–3.

1 piece of plastic film, Saran wrap, about 6×8 inches. See Lab Hint 4.

LAB HINTS

1. Selection of Sets of Solutions

Several sets of solutions are listed below in Lab Hint 2. It is not intended that any one student use all the sets described. Two more complicated sets (4 and 5) have been listed, but remember the purpose of the experiment is to help the student develop his powers of logical thinking. The method of analysis is of primary importance.

The set of solutions used first should be simple and should not include many combinations which form precipitates. The first three sets are simple. In the first, only one pair of ions precipitate, giving BaCrO_4 , and each other combination of spectator ions associated with BaCrO_4 checks out as being soluble. In the second set, there are two different precipitates, BaSO_4 and SrSO_4 . Each occurs twice. Each combination of spectator ions associated with these precipitates checks out as being soluble. In the third set, KCl , which is found once with a precipitate $\text{Fe}(\text{OH})_3$ and once with $\text{Co}(\text{OH})_2$, cannot be shown to be soluble. However, the possible combination of NaCl does check out and most students would associate KCl as being like NaCl and arrive at the correct conclusion. This situation provides opportunity to *test* their hypotheses.

2. Preparation of Sets of Solutions

Plastic, squeeze-type dropper bottles are ideal as containers for the solutions.

Check the solutions before the students use them. If the expected precipitates do not appear, increase the concentrations. The mass needed to prepare 100 ml of solution is included after the formula of each compound.

Set 1

0.1 M $\text{Ba}(\text{NO}_3)_2$ (2.6 g)	0.1 M K_2CrO_4 (1.9 g)
0.1 M BaCl_2 (2.4 g $\text{BaCl}_2 \cdot 2 \text{H}_2\text{O}$)	0.2 M NaNO_3 (1.7 g)
0.1 M Na_2CrO_4 (1.6 g)	0.2 M KCl (1.5 g)

(Four of the 15 combinations give ppts—all are BaCrO_4 .)

Set 2

0.1 M Na_2SO_4 (1.4 g anhydrous)	0.1 M MgCl_2 (2.0 g $\text{MgCl}_2 \cdot 6 \text{H}_2\text{O}$)
0.07 M $\text{Al}_2(\text{SO}_4)_3$ (4.7 g $\text{Al}_2(\text{SO}_4)_3 \cdot 18 \text{H}_2\text{O}$)	0.1 M $\text{Ba}(\text{NO}_3)_2$ (2.6 g)
0.1 M $\text{Sr}(\text{NO}_3)_2$ (2.1 g)	0.07 M AlCl_3 (1.7 g $\text{AlCl}_3 \cdot 6 \text{H}_2\text{O}$)

(Four of the 15 combinations give ppts—two are SrSO_4 ; two are BaSO_4 .)

Set 3

0.1 M FeCl_3 (2.7 g $\text{FeCl}_3 \cdot 6 \text{H}_2\text{O}$)	0.2 M NaOH (0.80 g)
0.1 M $\text{Co}(\text{NO}_3)_2$ (2.9 g $\text{Co}(\text{NO}_3)_2 \cdot 6 \text{H}_2\text{O}$)	0.2 M KOH (1.12 g)
0.1 M CoCl_2 (2.4 g $\text{CoCl}_2 \cdot 6 \text{H}_2\text{O}$)	0.2 M NaNO_3 (1.7 g)

(Six of the 15 combinations give ppts—two are $\text{Fe}(\text{OH})_3$; four are $\text{Co}(\text{OH})_2$.)

Two further suggestions are:

Set 4

0.1 M NiCl_2 (2.4 g $\text{NiCl}_2 \cdot 6 \text{H}_2\text{O}$)	0.2 M NaOH (as in set 3)
0.1 M MgCl_2 (2.0 g $\text{MgCl}_2 \cdot 6 \text{H}_2\text{O}$)	0.1 M $\text{Ba}(\text{OH})_2$ (saturated soln., 1.7 g)
0.1 M Na_2SO_4 (as in set 2)	0.1 M MgSO_4 (2.5 g $\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$)

(Seven of the 15 combinations give precipitates; one combination gives two.)

Set 5

0.1 M BaCl_2 (as in set 1)0.07 M $\text{Al}_2(\text{SO}_4)_3$ (as in set 2)0.1 M $\text{Sr}(\text{NO}_3)_2$ (as in set 2)0.1 M K_2CrO_4 (as in set 1)0.1 M Na_2CrO_4 (as in set 1)0.2 M AgNO_3 (3.4 g)

(Ten of the 15 combinations give precipitates.)

- Many teachers find it useful to have students do Set 1, and discuss it in detail before assigning another set. Good review is provided by having the different sets spread several days or even weeks apart.
- Saran wrap or other clear plastic film works very well. Drops of liquid placed on the plastic ball-up and the surface is easily blotted dry. A medium-dark colored background works best to give contrast for most of the precipitates. Use a piece of paper under the plastic film.

PRELAB DISCUSSION

Some students will need help in organizing a table and deciding how many combinations are to be tested. Students tend to show coefficients with formulas of ions when listing them in the table. That is, for MgCl_2 , many show $\text{Mg}^{2+} + 2 \text{Cl}^-$. Help them understand that coefficients are not needed. They tend to confuse rather than help.

Having recently done Experiment 15, you can point out that MgCl_2 gives Mg^{2+} ions and Cl^- ions rather than Mg^{2+} and Cl_2^{2-} ions. Solvents in which MgCl_2 dissolves show three times as much Δt_f as is shown by camphor or urea and half again as much as NaI or KI .

After the students have had a chance to prepare their data table, discuss the possible combinations with them and suggest that adding some of a solution to a sample of the same solution is not necessary.

Be careful not to impose your ideas or your methods of analysis on them—to you the experiment will seem very simple and obvious, but to the students it will be a useful and challenging experience. For those who need help in processing the data, do not give them more help than is necessary. Force them to try to figure it out on their own.

However, for your own peace of mind when grading the papers, or when discussion takes place, insist on consistency in labels on data tables. Have all students list ions in the same sequence along the horizontal axis, and in the opposite order along the vertical axis. Writing up an experiment with one of these sets is often a rather heavy homework assignment for many students.

PRECAUTIONS

Observe the usual laboratory precautions. Instruct the students on how they can avoid contaminating the solutions by not touching dropper bottles or medicine droppers to drops of other solutions.

POSTLAB DISCUSSION

One of the purposes of this experiment is to introduce *reacting species* and to use them in writing equations to represent chemical reactions. In addition, students should realize that the reason some substances precipitate and others do not often depends on concentration. We cannot predict which compounds will precipitate except by an accumulation of experience

and knowing the concentrations. There are no simple rules for prediction, but some of the trends are presented in Table 6-2, p. 97 of the Textbook.

Some of the students may be led to make generalizations that are too broad. We hope they will say something like, "When Ba^{2+} and SO_4^{2-} are present a precipitate forms." If grander generalizations are proposed suggest they do additional experiments to test their hypothesis.

SAMPLE DATA

For set 3, a student's record should look like this:

Solution	6 $\text{Na}^+, \text{NO}_3^-$	5 K^+, OH^-	4 Na^+, OH^-	3 $\text{Co}^{2+}, \text{Cl}^-$	2 $\text{Co}^{2+}, \text{NO}_3^-$	1 $\text{Fe}^{3+}, \text{Cl}^-$
1. $\text{Fe}^{3+}, \text{Cl}^-$	NR	ppt	ppt	NR	NR	
2. $\text{Co}^{2+}, \text{NO}_3^-$	NR	ppt	ppt	NR		
3. $\text{Co}^{2+}, \text{Cl}^-$	NR	ppt	ppt			
4. Na^+, OH^-	NR	NR				
5. K^+, OH^-	NR					
6. $\text{Na}^+, \text{NO}_3^-$						

NR means no reaction observed.

RESULTS OF PROCESSING THE DATA

- 1) Since the pairs of ions listed for each solution were present in the solution, you may assume that each precipitate formed was due to a new combination of these ions. For example, if you mix aqueous solutions of AgNO_3 and NaCl there are two new combinations of ions possible. The silver nitrate solution contains $\text{Ag}^+(\text{aq})$ and $\text{NO}_3^-(\text{aq})$. The sodium chloride solution contains $\text{Na}^+(\text{aq})$ and $\text{Cl}^-(\text{aq})$. Possible new combinations of these ions are AgCl and NaNO_3 .

Make a list of all possible new combinations of ions for each pair of solutions that produced a precipitate.

All possible new combinations:

$\text{Fe}(\text{OH})_3$ and KCl	KCl not eliminated; make further test
$\text{Fe}(\text{OH})_3$ and NaCl	NaCl eliminated by results on mixing 1 and 6
$\text{Co}(\text{OH})_2$ and KNO_3	KNO_3 eliminated by results on mixing 5 and 6
$\text{Co}(\text{OH})_2$ and NaNO_3	NaNO_3 is soluble (solution 6)
$\text{Co}(\text{OH})_2$ and KCl	KCl not eliminated
$\text{Co}(\text{OH})_2$ and NaCl	NaCl eliminated above

(See Lab Hint 2 for other possible new combinations listed with sets of solutions.)

- 2) Examine your data and propose explanations that will account for the fact that a precipitate was formed with some combinations and not with others.

Some explanations that have been suggested are:

Set 1 (a) Ba^{2+} precipitates with CrO_4^{2-} .

(b) If the charge of each ion is 2, then a precipitate results.

Set 2 Ba^{2+} and Sr^{2+} both precipitate with SO_4^{2-} .

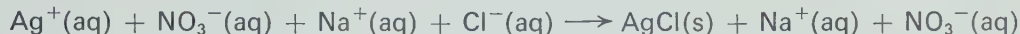
Set 3 (a) Fe^{3+} and Co^{2+} precipitate with OH^- .

(b) Elements not in the first column of the Periodic Table precipitate with OH^- .

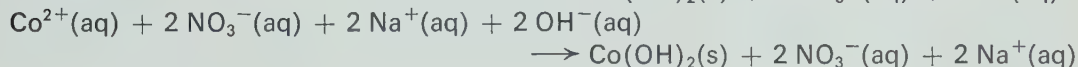
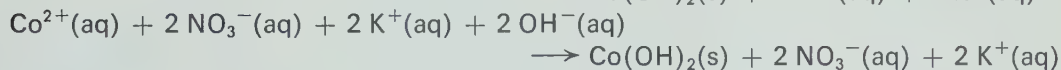
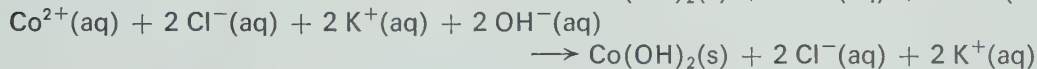
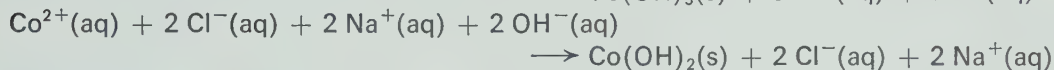
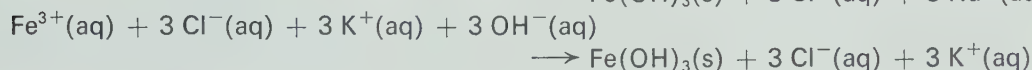
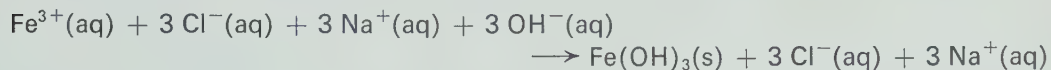
- 3) Examine your data and see if you can justify eliminating some of the combinations listed as possible precipitates.

Justification could be that a solution of the substance was used. For example, sodium nitrate cannot be one of the precipitates.

- 4) Write equations to indicate what you consider to have happened in each case in which there was a precipitate formed. Use ions to represent the species in the reacting solutions, but for those products that were precipitates write a formula for the compound. Place (aq) after those species in solution, and (s) after the precipitates. Be sure to write the equations so that both atoms and charge are conserved. For example:

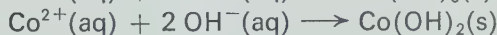
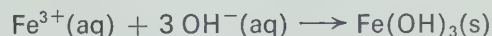


For Set 3 we have the following equations:



- 5) Rewrite these equations leaving out the ions not involved in the reaction. Such an equation, which shows only the predominant reacting species, is called a *net ionic equation*.

For Set 3 we have:



DEMONSTRATION 22

SOLVENT EFFECT ON CONDUCTIVITY

PURPOSE

To emphasize the role of the solvent.

TIMING

About 20 minutes during Section 6-8.

MATERIALS NEEDED

electrical conductivity apparatus. See Demonstration 21, p. 99.

NaCl(s)

NaCl, 1 *M*

AgNO₃(s). See Lab Hint 1.

AgNO₃, 0.1 *M*

HCl(g). See Lab Hint 2.

water

benzene

LAB HINTS

1. AgNO₃(s) in a crucible can be used year after year. Avoid overheating—just get it warm enough so that not too much solidifies on the electrodes during testing.
2. HCl(g) is easily obtained from a lecture bottle. If this is not available set up an HCl generator. Use a small amount of coarse rock salt and conc. H₂SO₄. The HCl produced is dry.

PROCEDURE

Have students record the results of testing: NaCl(s), NaCl 1 *M*, AgNO₃(s), AgNO₃(*l*), AgNO₃ 0.1 *M*, H₂O, H₂O into which HCl(g) is bubbling, benzene, and benzene into which HCl(g) is bubbling.

FOLLOW-UP

Note the solids did not conduct. Ask students for explanation.

Ions are not mobile enough. AgNO₃(*l*) did conduct—ions are mobile. Apparently water will ionize HCl(g), but benzene won't. If there is a question about the HCl(g) dissolving in the benzene, slowly bubble the gas into fresh benzene in a tall cylinder. As the bubbles rise, they get smaller.

DEMONSTRATION 23
CONDUCTIVITY OF ACIDS, BASES, AND SALTS

PURPOSE

To show the conductivity of solutions of a variety of acids, bases, and salts.

TIMING

About 30 minutes during Section 6-9.

MATERIALS NEEDED

conductivity apparatus, Demonstration 21, p. 93

0.1 *M* solutions of HCl, H₂SO₄, HC₂H₃O₂, H₃PO₄, NaOH, KOH, NH₄OH, NaCl, KHSO₄, K₂SO₄, NH₄C₂H₃O₂, CaCl₂, AgNO₃

saturated solutions of Ca(OH)₂, Ba(OH)₂, respectively 0.02 *M* and 0.1 *M*

20 beakers, 50 ml

PROCEDURE

Have the students record the results of the conductivity tests.

Points to emphasize:

- 1) light is dimmer with $\text{HC}_2\text{H}_3\text{O}_2(\text{aq})$ than with $\text{HCl}(\text{aq})$. The former is the weaker acid.
- 2) $\text{NH}_4\text{OH}(\text{aq})$ produces a weaker glow (is a weaker base) than $\text{NaOH}(\text{aq})$ or $\text{KOH}(\text{aq})$.
- 3) $\text{NH}_4\text{C}_2\text{H}_3\text{O}_2(\text{aq})$ produces as bright a glow as the other solutions. Dissolved salts are highly ionized.

Experiment 18

Types of Solutions

PURPOSE AND SUMMARY

To study unsaturated, saturated, and oversaturated solutions. The heat effects of dissolving and crystallization are noted.

The student makes solutions using sodium acetate. He seeds an oversaturated solution to observe heat effects and spectacular crystallization.

TIMING

At the end of Chapter 6. About 30 minutes are needed in the lab.

MATERIALS NEEDED

per student

2 test tubes, 18×150 mm
 1 beaker, 250 ml
 1 test tube holder
 thumb protectors.
 See Lab Hint 5, Expt. 16, p. 88 in this Guide.

per class

burners, Bunsen
 test tube racks
 bottles of solid $\text{NaC}_2\text{H}_3\text{O}_2 \cdot 3 \text{H}_2\text{O}$. See Lab Hint 1.
 plastic teaspoons for above bottles

LAB HINTS

1. Hypo (sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5 \text{H}_2\text{O}$) is also cheap and satisfactory.
2. If you wish to salvage the crystals, have students pour the warm solutions in a plastic lined box. When the water evaporates, crush the solid and store.

PRELAB DISCUSSION

This experiment is simple and requires little preparation. Tell the students how you wish them to dispose of the crystals. See Lab Hint 2.

PRECAUTIONS

Observe the usual lab precautions.

POSTLAB DISCUSSION

Go over student answers to the questions. Emphasize the reversibility of the heat effect: endothermic during solution, exothermic during crystallization. It is worth pointing out that oversaturated solutions are rarely found in nature because they are too unstable.

SAMPLE DATA AND EXTENDED WRITE-UP

The questions in the PROCEDURE are meant to be rhetorical. However answers are given below.

- a. The crystals dissolved, the solution was not saturated. The solution might now be saturated. But the odds are against it.
- b. The temperature dropped. The added crystal will likely not dissolve. The solution is now saturated.

PROCESSING THE DATA

- 1) What experiment would you do at the end of step c to determine if the solution produced was saturated? Explain why you think it was or was not saturated.

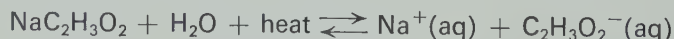
Add another crystal. Note whether it dissolves. If it does, the solution is unsaturated. If it does not, the solution is saturated. It was likely unsaturated because heating was continued a bit after the last solid dissolved. Usually the solubility of a solid in a liquid increases with temperature.

- 2) Because of what happened in step e, do you think the solution produced in step (d) can be adequately described as saturated? Explain.

No, the solution was more than saturated. It contained more solid than it would normally hold. A speck of dust, an added crystal, vibration or agitation caused the release of considerable solid.

- 3) Explain the observed temperature changes as NaAc dissolved and as it crystallized out of solution.

As the crystals dissolved, heat was absorbed. As crystals formed, heat was liberated. These changes can be represented:



If energy is conserved, then we expect the heat absorbed during solution to be released during crystallization.

- 4) List four *different* ways of making a saturated solution.

- 1) Dissolve as much solid as possible with agitation.
- 2) Cool an unsaturated solution.
- 3) Seed an oversaturated solution.
- 4) Evaporate some liquid from an unsaturated solution.
- 5) Combine two different solutions to form a precipitate, e.g. AgNO_3 and NaCl solutions.

Order Among Atoms



The Periodic Table expresses many regularities ; this is the theme of the chapter. The main laboratory information comes from the film CHEMICAL FAMILIES but there are two experiments dealing with qualitative analysis.

Schedule of Experimental Work

TEXT SECTION	EXPERIMENTAL WORK	FOR DETAILS SEE PAGE
7-1, 7-2	Expt. 19	104
	Expt. 20	106
7-3 to 7-5		

Experiment 19

Introduction to Qualitative Analysis

PURPOSE AND SUMMARY

To serve as an introduction to qualitative analysis. To develop a scheme by which an unknown solution may be identified as being like one or more of the solutions tested.

The student tests the behavior of four different solutions with three different reagents. After charting their characteristic reactions he compares the behavior of an unknown solution to that of the four solutions. From the observations using all three reagents he is able to conclude which of the tested solutions the unknown solution is *like*.

TIMING

This experiment is recommended during Chapter 7 because there are no other student experiments available that are closely related to the textual material.

If one set of reagents is provided for each pair of students, about 20 to 30 minutes are required.

MATERIALS NEEDED

per student or pair

1 piece of plastic film, Saran wrap about 6×8 inches (one piece per student)

1 set of solutions and reagents in dropper bottles

Solution	Substance (amount needed per 100 ml)
1	NaCl (0.6 g)
2	Na ₂ SO ₄ (1.4 g or 3.2 g Na ₂ SO ₄ · 10 H ₂ O)
3	K ₂ CrO ₄ (1.9 g)
4	K ₄ Fe(CN) ₆ · 3 H ₂ O (4.2 g) or K ₄ Fe(CN) ₆ · 6 H ₂ O (4.8 g)
Reagents	Substance (amount needed per 100 ml)
A	AgNO ₃ (1.7 g)
B	Ba(NO ₃) ₂ (2.6 g)
C	Zn(NO ₃) ₂ · 6 H ₂ O (3.0 g)

unknown solutions. See Lab Hint 2.

LAB HINTS

1. Small plastic squeeze bottles that deliver drops are ideal for this experiment. Store silver nitrate in brown-colored bottles.

2. The single unknown solutions can be obtained from the stock solutions: 1, 2, 3, and 4. However, for unknowns containing two or more substances, the concentration of ions must be the same as in the test solutions. If you mix Solutions 1 and 2, the anions will be diluted and reactions may not occur. For example, to prepare an unknown solution containing two of the substances, NaCl and Na₂SO₄, weigh out 0.6 g NaCl and 3.2 g Na₂SO₄ · 10 H₂O and dissolve both amounts in 100 ml of solution. For triple or quadruple unknowns, add the indicated amounts of three substances or four substances to 100 ml of solution.

PRELAB DISCUSSION

Discuss the introduction and procedure with the students. Make sure they understand the procedure and the technique of analysis. They used the drop test once before in Experiment 17. They may, however, need to be reminded to keep the solutions and reagents free from contamination. Many students have a strong tendency toward touching the drops of one reagent or solution with the dropper or nozzle of another container.

Try to develop the spirit of a detective game for the experiment. Encourage them to look for the characteristic behavior and to be sure to keep good records of their observations.

PRECAUTIONS

Observe usual laboratory precautions. Remind students to keep solutions free from contamination.

POSTLAB DISCUSSION

Help the students realize that an organized scheme of analysis is essential. They should realize that organization rather than hit-or-miss mixing is an efficient use of time and effort, and is necessary for more complex analysis. Point out that the order of testing is not of primary importance—that different students are expected to use different procedures. There is no one best way to proceed. But each should, nevertheless, have an organized scheme of analysis and should be able to properly analyze an unknown.

SAMPLE DATA

Solutions	Reagents		
	A (Ag ⁺)	B (Ba ²⁺)	C (Zn ²⁺)
1 (Cl ⁻)	white ppt	no reaction	no reaction
2 (SO ₄ ²⁻)	no reaction	white ppt	no reaction
3 (CrO ₄ ²⁻)	brick-red ppt	yellow ppt	no reaction
4 (Fe(CN) ₆ ⁴⁻)	white ppt	no reaction	white ppt (gelatinous)

Data for Two-substance Unknown

Mixed Solution	Reagents		
	A	B	C
1 and 2	white ppt	white ppt	no reaction
1 and 3	red ppt at first, changes to white	yellow ppt	no reaction
1 and 4	white ppt	no reaction	white ppt (gelatinous)
2 and 3	brick-red ppt	creamy-yellow ppt	no reaction
2 and 4	white ppt	white ppt	white ppt (gelatinous)
3 and 4	brick-red changes to yellow or white	yellow ppt	white ppt (gelatinous)

For the mixed unknowns, difficulty may be encountered with mixtures of 1 with 4, and 2 with 3. If the student knows that the solution contains two unknown substances, he can deduce that solution 1 could be present with 4. That is, he will get the same reactions with solutions 1 and 4 as with 4 alone. He can also deduce that the white precipitate from solution 2 could be present with 3 but masked by the yellow precipitate from 3.

Experiment 20

Qualitative Analysis—Relative Solubilities

PURPOSE AND SUMMARY

To study the slight differences in solubility of some second-column metallic compounds and to use these differences to devise a qualitative analysis scheme to establish the presence of one of these cations in an unknown.

The student tests four second-column cations with five anions. The carbonate precipitates are given a flame test.

TIMING

About 25 minutes are needed to complete Steps a, b, and c. After the student writes up the experiment, and devises a system of analysis, about 20 minutes are needed for an unknown. The experiment fits any place in Chapter 7.

MATERIALS NEEDED

per student

- 1 piece of plastic film, 6 × 8 inches
- 3 test tubes, 13 × 100 mm

Reagents (in dropper bottles)

2 *M* $(\text{NH}_4)_2\text{CO}_3$ with $\text{NH}_3(\text{aq})$ (192 g $(\text{NH}_4)_2\text{CO}_3$ dissolved in water and 80 ml 15 *M* $\text{NH}_3(\text{aq})$ /liter)

0.5 *M* K_2CrO_4 (97 g/liter)

0.2 *M* $(\text{NH}_4)_2\text{C}_2\text{O}_4$ (28.4 g $(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ /liter)

1 *M* $(\text{NH}_4)_2\text{SO}_4$ (132 g/liter)

6 *M* $\text{NH}_3(\text{aq})$ (400 ml conc. $\text{NH}_3(\text{aq})$ /liter)

6 *M* HCl (515 ml conc. HCl /liter)

Test Solutions (in dropper bottles) :

0.1 *M* $\text{Ba}(\text{NO}_3)_2$ (26 g/liter)

0.1 *M* $\text{Sr}(\text{NO}_3)_2$ (21 g/liter)

0.1 *M* $\text{Ca}(\text{NO}_3)_2$ (24 g $\text{Ca}(\text{NO}_3)_2 \cdot 4 \text{H}_2\text{O}$ /liter)

0.1 *M* $\text{Mg}(\text{NO}_3)_2$ (26 g $\text{Mg}(\text{NO}_3)_2 \cdot 6 \text{H}_2\text{O}$ /liter)

Unknowns containing two cations should be made from equal volumes of 0.2 *M* solutions.

For confirmatory tests :

platinum or nichrome flame-testing wire. See Lab Hint 1.

lab burner ; 5 ml 12 *M* HCl

LAB HINTS

1. A small loop at the end of the wire is often used but is not essential. If you prefer that there be a loop in the test wire, you should make it before giving the wire to a student. The student should be asked not to unbend it. If the wire is imbedded in glass, the student must be cautioned not to touch the hot glass to the HCl or test solutions.

It is very difficult to get rid of the short burst of yellow produced by sodium contamination. This is mentioned in the Laboratory Manual, but more discussion may be needed.

The emission of energy of specific wavelengths by atoms should be discussed and the relationship of flame tests to more complete spectroscopic examination pointed out. Remind the student of the discussion in Chapter 9.

PRELAB DISCUSSION

Caution the students about contaminating the solutions by touching one solution with the tip of a dropper from another.

Create an atmosphere of research. Emphasize the importance of identifying their unknown correctly, and on the basis of the evidence they themselves gathered.

Mention that the 1 ml of solution needed in Step b can be estimated. Using a graduated cylinder only introduces another chance for contamination.

PRECAUTIONS

Observe the usual lab precautions. The concentrated HCl should be handled with special care. Wear safety glasses.

POSTLAB DISCUSSION

Emphasize that a systematic arrangement of knowledge gained from experiments is a very powerful basis from which accurate predictions can be made.

Typical student results are :

	CO_3^{2-}	CrO_4^{2-}	SO_4^{2-}	$\text{C}_2\text{O}_4^{2-}$	$\text{NH}_3(\text{aq})$	Flame color
Mg^{2+}	NR	NR	NR	NR	slight white ppt	none
Ca^{2+}	white ppt	NR	NR	white ppt	NR	orange red
Sr^{2+}	white ppt	NR	white ppt	white ppt	NR	scarlet
Ba^{2+}	white ppt	yellow ppt	white ppt	white ppt	NR	pale green

For a mixture of cations, the flame test will first reveal the red-orange from Ca^{2+} , then a flash of green from Ba^{2+} , and finally the crimson from Sr^{2+} . Be sure the concentrations for unknown solutions are the same as those for the test solutions.

The student should have no difficulty identifying an unknown containing one of these cations. Those doing the optional unknown may need help in developing a scheme of analysis. One scheme that the student might work out follows :

To 5 ml unknown :

Add 1 ml $\text{CrO}_4^{2-}(\text{aq})$, yellow ppt indicates Ba^{2+} .

Centrifuge or filter to separate. To the filtrate, add 1 ml $\text{SO}_4^{2-}(\text{aq})$, white ppt indicates Sr^{2+} .

Centrifuge or filter to separate. To the filtrate, add 1 ml $\text{C}_2\text{O}_4^{2-}(\text{aq})$, white ppt indicates Ca^{2+} .

Centrifuge or filter to separate. To the filtrate, add 1 ml $\text{NH}_3(\text{aq})$, white ppt indicates Mg^{2+} .

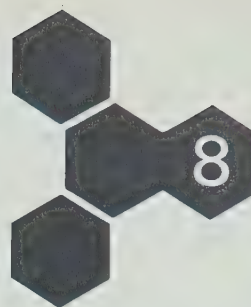
PROCESSING THE DATA

- 1) a. Which carbonate of the second column metals has the greatest solubility? Which ones have similar solubility?
b. Describe a possible separation of the cations based upon the above differences.
 - (a) MgCO_3 has the greatest solubility; the others have similar solubilities, but lower than that for magnesium.
 - (b) The Mg^{2+} ion could be separated from the other second column cations by precipitating their carbonates and filtering or centrifuging.
- 2) a. Which chromate of the metals is the least soluble?
b. How can this difference in solubility be used in an analytical separation of a solution containing 0.1 M solutions of Sr^{2+} and Ba^{2+} ?
 - (a) The least soluble chromate is BaCrO_4 .
 - (b) The Ba^{2+} could be precipitated from a 0.1 M solution and be separated from Sr^{2+} by filtration.
- 3) With which of the anions does the Mg^{2+} ion have the lowest solubility?

Mg^{2+} has the lowest solubility as $\text{Mg}(\text{OH})_2$. In all other cases the Mg^{2+} compound is more soluble than that of the other alkaline earths.
- 4) Which oxalate of these metals is the most soluble?

The most soluble oxalate is magnesium oxalate.

The Structure of the Atom



This chapter is difficult to support with experimental work by the students because of the complex nature of the apparatus required. Our picture of the atom comes from observations of gas discharge tubes, spectra, mass spectrographs, and alpha-particle scattering. Some of these can be done qualitatively as in Demonstration 25 on the spectroscope. However, most of the evidence for the conclusions must be accepted by the student from the descriptions given in the text.

Schedule of Experimental Work

TEXT SECTION	EXPERIMENTAL WORK	FOR DETAILS SEE PAGE
8-1	Expt. 21	110
	Demo. 24	114
	Demo. 25	116
8-2 to 8-8		
	Expt. 22	112

Experiment 21

Construction of a Logical Model

PURPOSE AND SUMMARY

To help build the *model* concept as used in science. To provide experience in the process of inferential thinking as illustrated in the *garbage collector analogy*. (See Teacher's Guide, p. 131.)

Each student is given a sealed box which contains a hidden object or objects. Without opening the box, they make as many observations as possible. They obtain as much information as they can in order to construct a mental model which will account for *all* the observed properties of their system.

TIMING

The experiment should come early in Chapter 8, following a discussion of the *garbage collector analogy*.

The experiment requires parts of two periods. One to demonstrate what is to be done and to make observations of the systems. Part of another period is needed to consider other students' analyses of the systems, and for discussion.

MATERIALS NEEDED

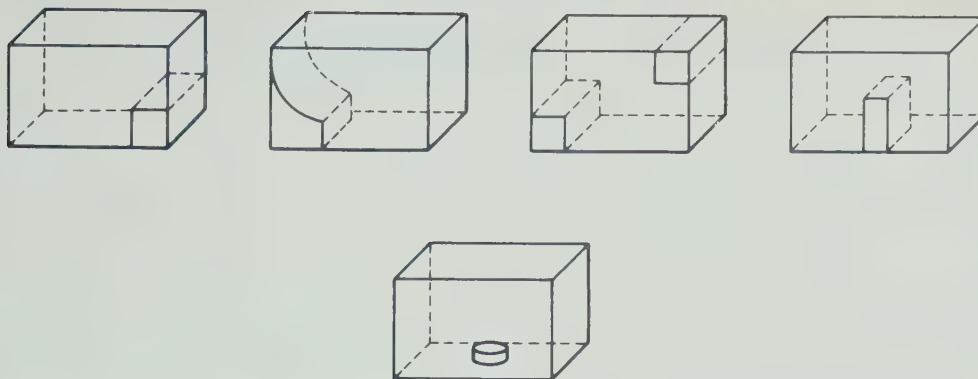
per student

1 *black box* system. See Lab Hints.

LAB HINTS

1. Prepare one class set of *black boxes*. Number each one uniquely. Many classes can use the same set.
2. Shoe Box Method
Shoe boxes or cigar boxes are suitable. For most students the objects in the boxes should be simple. Use a variety of objects in the boxes that can be found around the laboratory such as: a rubber or cork stopper, a test tube, rubber tubing, a watch glass, a metal washer, a pencil, a nut, a bolt, etc. Seal each box after placing an object in it. Some interesting, but difficult, ones are: (a) two small rubber balloons, containing some water, and tied together; (b) a spool threaded on a string held taut by being glued to opposite ends of the box.
3. Marble Probe Method

Glue an object to the inside of a shoe box as in the drawings below. Place a marble in the box with the object and fasten the lid in place. Have the students use the marble as a probe to "build a model" of what the object inside must be like. The concealed objects can be cut out of wood.



PRELAB DISCUSSION

Show one of the *black box* systems to the class before passing them out. (See Lab Hints.) Make a few simple observations to give the students an idea of what they are to do. Describe your observations and try to get the students involved in making inferences from the observations. Select inferences which lead to hypotheses and test them by experiment. For example, as a box is tilted it is observed that the object slides along the bottom. It can be inferred from this observation that the object may have at least one flat side. Additional experiments, such as tilting the box, may confirm this hypothesis. On the basis of experimental evidence, many and repeated observations, accept or reject the hypothesis tested.

Be sure to point out and emphasize a definite procedure—observation leads to inferences; inferences lead to experiment; experiment and additional observations lead to other inferences and to a model. Finally, suggest the students do the same with the black boxes given to them. It is not the purpose of the experiment to identify the concealed object, rather, to form a model to account for observed properties.

Each student should prepare a report which includes his observations, inferences, experiments, and conclusions leading to his mental model. The report should also include the number of the system he used. At the end of the observing period, collect all reports. At the end of the day assemble them according to black box number. You may wish to clip together all the reports for box #1, #2, The students may then read many reports on the same object.

PRECAUTIONS

Remind the students they are not to open the box or deform it in any way.

POSTLAB DISCUSSION

Distribute the reports along with the matching box system. Each student should receive the same system he investigated, along with all of the reports for that system. Give them 5 or 10 minutes to study the reports and do additional experiments. Call on several individuals to give summary reports, and present an argument in defense of their proposed model. Have them justify their position concerning conflicting models proposed by others who have observed the same system. The models thus deduced should be accepted as the best they can do with the experiments available to them. The boxes should remain unopened. This corresponds to our inability to look into atomic systems and *see real* answers. Our inferences are the real answers as far as we know them.

Experiment 22

Qualitative Analysis: Ag^+ , Hg_2^{2+} , and Pb^{2+}

PURPOSE AND SUMMARY

To develop a scheme of analysis using solubility information and techniques acquired in Experiments 19 and 20.

The student first is given the opportunity to become familiar with characteristic reactions used to identify silver, mercurous, and lead ions in aqueous solutions. After making these observations he is required to devise a method of analysis in order to identify the presence of one or more of these ions in an *unknown solution*.

TIMING

The experiment can be done anytime following Experiments 19 and 20.

With a set of reagents and solutions for each pair of students, one period should be sufficient—including the determination of one *unknown solution*.

MATERIALS NEEDED

per student or pair

1 beaker, 250 ml
6 test tubes, 13 × 100 mm
1 burner, ring stand, ring, and wire gauze
1 centrifuge (if available)
1 set of solutions and reagents in dropper bottles
Solutions (amounts are per 100 ml of solution)

1 ml 0.1 M AgNO_3 (1.7 g)
1 ml 0.1 M $\text{Hg}_2(\text{NO}_3)_2$ (5.6 g $\text{Hg}_2(\text{NO}_3)_2 \cdot 2 \text{H}_2\text{O}$)
1 ml 0.1 M $\text{Pb}(\text{NO}_3)_2$ (3.3 g)
Reagents (amounts are per 100 ml of solution)
1 ml 6 M HCl (51.3 ml concentrated)
1 ml 0.1 M K_2CrO_4 (1.9 g)
6 ml 6 M $\text{NH}_3(\text{aq})$ (40 ml concentrated NH_4OH)
3 ml 6 M HNO_3 (38.4 ml concentrated)

LAB HINTS

1. Solutions and reagents should be made available in dropper bottles or plastic squeeze bottles. One set per pair is ideal, but one for four students will do.
2. *Unknown solutions* containing more than one cation should be made up so that the cation concentration is the same as in the test solutions (0.1 M). Prepare the *unknown solutions* by adding the indicated amount of two or three solids as given below:

Unknown Solutions Containing Two Cations (amounts given are per 100 ml of solution)

Ag^+ and Hg_2^{2+} [1.7 g AgNO_3 + 5.6 g $\text{Hg}_2(\text{NO}_3)_2 \cdot 2 \text{H}_2\text{O}$]

Ag^+ and Pb^{2+} [1.7 g AgNO_3 + 3.3 g $\text{Pb}(\text{NO}_3)_2$]

Hg_2^{2+} and Pb^{2+} [5.6 g $\text{Hg}_2(\text{NO}_3)_2 \cdot 2 \text{H}_2\text{O}$ + 3.3 g $\text{Pb}(\text{NO}_3)_2$]

Unknown Solution Containing All Three Cations

[1.7 g AgNO_3 + 5.6 g $\text{Hg}_2(\text{NO}_3)_2 \cdot 2 \text{H}_2\text{O}$ + 3.3 g $\text{Pb}(\text{NO}_3)_2$]

PRELAB DISCUSSION

None is needed other than to mention this experiment follows the same general pattern as in Experiments 19 and 20. Students should be able to proceed on their own.

PRECAUTIONS

Observe the usual laboratory precautions.

POSTLAB DISCUSSION

Some general observations and information :

Chloride Precipitates

$\text{Hg}_2\text{Cl}_2(\text{s})$ is very bulky

$\text{PbCl}_2(\text{s})$ is fine and settles quickly

$\text{AgCl}(\text{s})$ is bulky, and settles slowly

Solubilities of Chlorides grams per 100 ml water

Hg_2Cl_2 0.00021 (18°C) and 0.001 (43°C)

PbCl_2 0.99 (20°C) and 3.34 (100°C)
 AgCl 0.000089 (10°C) and 0.0021 (100°C)
 K_{sp} at 25°C

AgCl 1.7×10^{-10}

Hg_2Cl_2 2×10^{-18}

PbCl_2 1×10^{-4}

PbCrO_4 2×10^{-16}

SAMPLE DATA

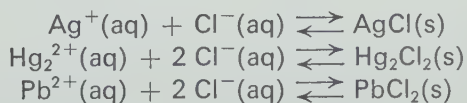
Treatment	Ag^+	Chloride Precipitate of Hg_2^{2+}	Pb^{2+}
warmed in boiling water	remains	remains	dissolves
CrO_4^{2-} added to hot solution	no change	no change	yellow ppt
6 M NH_3 added	dissolves	turns black	remains
6 M HNO_3 added	white ppt	no change	remains

OPTIONAL EXERCISES

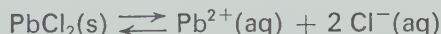
- 1) Write equations for all reactions occurring in this experiment. You may wish to postpone this assignment until after a study of equilibrium in Chapter 13.

The student will probably need help with two of the reactions: the formation of silver ammonia complex and mercurous chloride reaction with ammonia.

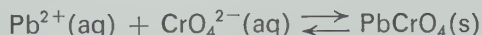
Reactions in Steps a, b, and c.



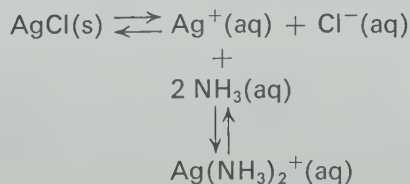
When $\text{PbCl}_2(\text{s})$ is warmed in water it dissolves.



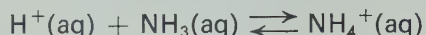
Chromate precipitates with Pb^{2+}



Reactions in Step e.

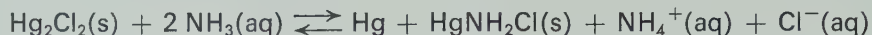


Addition of $\text{NH}_3(\text{aq})$ causes $\text{AgCl}(\text{s})$ to dissolve forming the complex ion, $\text{Ag}(\text{NH}_3)_2^+(\text{aq})$. Adding $\text{HNO}_3(\text{aq})$ reverses this reaction by reacting with $\text{NH}_3(\text{aq})$, releasing $\text{Ag}^+(\text{aq})$.

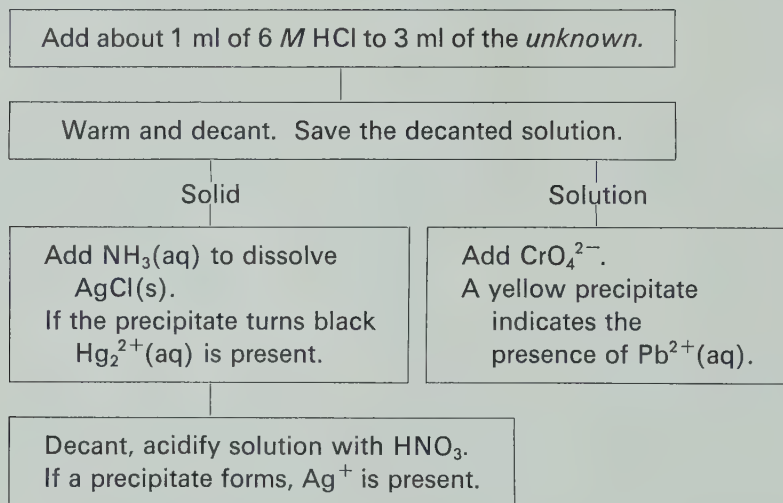


As the concentration of $\text{Ag}^+(\text{aq})$ increases $\text{AgCl}(\text{s})$ precipitates.

When ammonia is added to $\text{Hg}_2\text{Cl}_2(\text{s})$, a black substance, Hg , forms along with a white precipitate.



- 2) Construct a flow diagram which summarizes the steps you would use to analyze an *unknown* solution containing all three cations.



DEMONSTRATION 24

SPECTRA AND ABSORPTION

PURPOSE

To demonstrate the visible spectrum and to introduce the idea of absorption.

TIMING

About 15 minutes during Section 8-1.

MATERIALS NEEDED

1 slide projector

1 diffraction grating. See Lab Hint 1.

1 set of slides. See Lab Hint 2.

LAB HINTS

1. A good transmission diffraction grating, as in the Bausch and Lomb Educational Absorption Spectra Kit. It will give a 3-foot wide spectrum at 25 feet. Poorer replica diffraction gratings give poorer spectra. A glass prism will give a one-foot spectrum.
2. Suitable slides can be prepared using the following materials:

35 mm slide mount from the local photographic supply store or;
 #3i 35 mm SF frames from Porter Mfg. and Supply Co.

2215 No. Chico
 So. El Monte, Calif. 91733

Aluminum foil

Color filter sheets. Many kinds will do. Those from Edmund Scientific Co., Barrington, N. J. 08007 work well. For 44 different colors each $1'' \times 4''$, order #40675.

For preparation, cut a piece of aluminum foil as shown in Figure 22. Make one frame with no color filter. The following filter numbers (from the Edmund Scientific Set) produce these results.

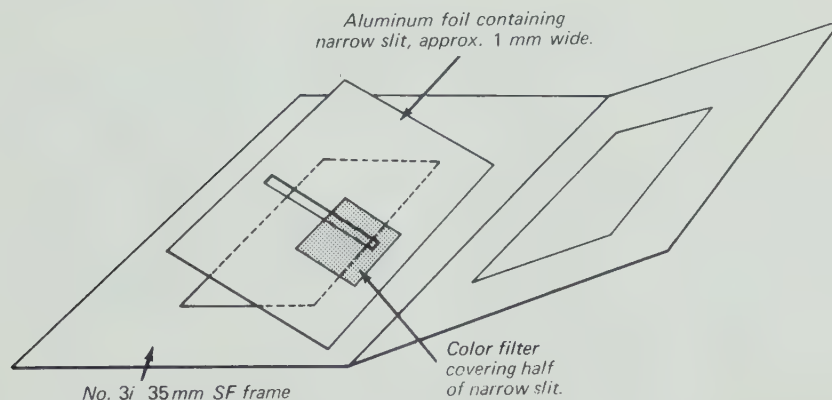


Figure 22 Slide for absorption spectra.

#821	red only
874	green, trace red and blue
866	blue with a trace of green
809	cuts out blue (it is yellow)
817	cuts out most of the green and blue (it is orange)
832	cuts out green (it is magenta)

CLASSROOM PRESENTATION

Set up a 35 mm slide projector and screen in the usual way. Position the grating a few inches in front of the projector so that the light goes through it. Adjust the angle until a spectrum appears on the screen.

Show the slide with no filter to give the full spectrum. Then use a red filter frame. The light that does not pass through the red filter produces the visible spectrum just as the blank slide did. The light that passes through the filter contains only red light. The other colors are absorbed by the pigments in the filter.

Then use a frame with a green filter. The light passing through it contains green light with only a trace of red and blue. Proceed in this way. Each time, a complete spectrum will be presented along with a partial one resulting from the interaction of the filter with the white light.

FOLLOW-UP

The general point to develop is that although all visible frequencies enter the colored filters, only certain ones come out. The rest are absorbed by the molecules of pigment. How does the pigment absorb only certain frequencies? A model to explain this will be part of Chapter 9.

DEMONSTRATION 25

LINE SPECTRA

PURPOSE

To demonstrate the line spectra of hydrogen and other gaseous elements.

TIMING

About 15 minutes are needed during Section 8-1.

MATERIALS NEEDED

- a spectroscope. See Lab Hint 1.
- a source of spectra. See Lab Hint 2.

LAB HINTS

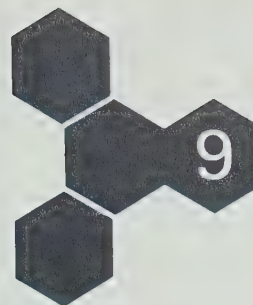
1. The spectroscope can range from an elaborate instrument borrowed from the Physics Lab, to a simple piece of diffraction grating costing a few cents.
2. Sources vary widely but a few simple ones are:
 - a. Hydrogen (or other) discharge tube energized by a spark coil or a more specialized power supply. Feeding too much current through a discharge tube shortens its life.
 - b. A lab burner equipped with an asbestos collar touching the flame. Moistening this collar with a salt solution gives a suitable spectrum.
 - c. An uncoated fluorescent tube (Macalaster #1328) is a good source of the mercury spectrum.

CLASSROOM PRESENTATION

Darken the room to avoid the continuous spectrum of sunlight. Have students examine several different spectra so they will understand hydrogen is not the only element with a line spectrum.

Raise the question: What is there about these elements that causes them to give out light of only certain wavelengths?

Models for Atomic Structure



This chapter uses the spectrum of atomic hydrogen to present the orbital model of electron arrangement. The text material comes from spectroscopic results and mathematical equations which cannot be studied in the high school laboratory. Therefore, we have provided two experiments of a descriptive type. One, on analysis of anions, will give you a chance to emphasize some reactions and show how differences in behavior lead to analytical procedures. The other experiment places many reactions before the student. These include reaction types that will be studied in the following chapters.

Schedule of Experimental Work

TEXT SECTION	EXPERIMENTAL WORK	FOR DETAILS SEE PAGE
9-1 to 9-5		
9-6	Demo. 26	118
9-7 to 9-12		

The experiments are not closely related to the Textbook sections.

Expt. 23	118
Expt. 24	121
Demo. 27	126

DEMONSTRATION 26

MODELS OF p ORBITALS

PURPOSE

To show a model of the p orbitals.

TIMING

About 2 minutes during Section 9-6.

MATERIALS NEEDED

A great variety of materials can be used. Perhaps the simplest is six fairly spherical rubber balloons. Tie the inflated balloons together in pairs of a similar color which is different from that used for the other pairs. Connect the pairs so they lie along the three perpendicular axes. They will tend to assume this position.

A more durable set can be made from Styrofoam eggs. See p. 29 for source.

PROCEDURE

Show the set of orbitals, emphasizing that each orbital consists of two lobes (shown by the similar colors). The entire set contains three p orbitals. Comment on their spatial arrangement. Each orbital of two lobes can accommodate up to two electrons. The resulting electron arrangements are *not* flat, or two dimensional, as the Bohr model can suggest.

Experiment 23

Analyzing for Anions

PURPOSE AND SUMMARY

Various anions (SO_4^{2-} , CO_3^{2-} , Cl^- , and I^-) are reacted with Ba^{2+} and Ag^+ . Precipitates are tested with H^+ and $\text{NH}_3(\text{aq})$. The student devises a scheme which can be used to identify an unknown which contains one or more of the four anions above. He also confirms some of the "solubility rules" given in Chapter 6.

TIMING

Steps a–e can be done easily in 20 minutes. Step f (develop a scheme of analysis) could be a homework assignment. "Trial unknowns" and teacher unknowns require 15 minutes. Anytime during Chapter 9 is satisfactory.

MATERIALS NEEDED**per student**

plastic film. See Lab Hint 1.
glass stirring rod, 2 mm $\times$ 10 mm
approximately 2 ml of each, except 10 ml of HNO_3 , in dropper bottles
 AgNO_3 , 0.1 *M* (1.7 g/100 ml)
 $\text{Ba}(\text{NO}_3)_2$, 0.1 *M* (2.6 g/100 ml)
 HNO_3 , 1.0 *M* (6.4 ml conc. reagent/100 ml)
 $\text{NH}_3(\text{aq})$, 6 *M* (40 ml conc. reagent/100 ml). See Lab Hint 2.
 Na_2CO_3 , 0.5 *M* (6.24 g $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$ /100 ml)
 NaCl , 0.1 *M* (0.6 g/100 ml)
 NaI , 0.1 *M* (1.5 g/100 ml)
 Na_2SO_4 , 0.1 *M* (1.4 g anhydrous Na_2SO_4 /100 ml)
approximately 10 ml of 0.2 *M* solutions of
 Na_2CO_3
 NaCl
 NaI
 Na_2SO_4
See Lab Hint 3.

LAB HINTS

1. Household plastic wrapping film is satisfactory. Each student needs a piece about 6" $\times$ 6". Glass plates will also work.
2. The $\text{NH}_3(\text{aq})$ should be fresh. Old solutions often have dissolved CO_2 , which gives carbonate precipitates.
3. Unknowns containing two anions should have the same anion concentration used in the student's experiment. It is difficult to detect both Cl^- and I^- in the same unknown.

PRELAB DISCUSSION

Little is needed. Experiments 19, 20, and 22 provide adequate background.

PRECAUTIONS

Exercise the usual lab precautions. HNO_3 still stains fingers. Wear safety glasses and aprons.

POSTLAB DISCUSSION

Much can be discovered about nature with a few inexpensive chemicals and dropper bottles. Prospectors often perform in the field the kind of tests developed in this experiment.

SAMPLE DATA

Results

Cation Added	$\text{SO}_4^{2-}(\text{aq})$	Anion in Solution Tested		$\text{I}^-(\text{aq})$
		$\text{CO}_3^{2-}(\text{aq})$	$\text{Cl}^-(\text{aq})$	
$\text{Ba}^{2+}(\text{aq})$				
$\text{H}^+(\text{aq})$ causes barium ppt to	ppt (white) NR	ppt (white) dissolve, bubbles	NR —	NR —
$\text{Ag}^+(\text{aq})$				
$\text{H}^+(\text{aq})$ causes silver ppt to	NR —	ppt (white-brown) dissolve, bubbles	ppt (white) NR	ppt (yellow) NR
$\text{NH}_3(\text{aq})$ causes silver ppt to	—	dissolve	dissolve	NR

Many possible schemes of analysis can be devised using these reagents. It is important to encourage a logical development and originality in approach.

EXAMPLES

1. Precipitate formation with Ba^{2+} shows either SO_4^{2-} or CO_3^{2-} :

A precipitate that dissolves with H^+ indicates CO_3^{2-} .

A precipitate that does not dissolve with H^+ indicates SO_4^{2-} . Partial dissolving is often difficult to recognize. The precipitate might contain both BaCO_3 and BaSO_4 .

2. Formation of a precipitate with Ag^+ indicates CO_3^{2-} , Cl^- , or I^- :

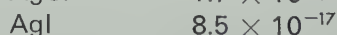
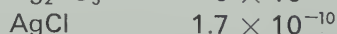
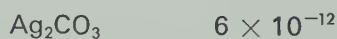
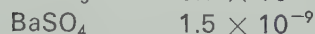
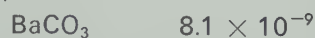
A precipitate that does not dissolve in $\text{H}^+(\text{aq})$ or $\text{NH}_3(\text{aq})$ indicates I^- (or a mixture).

A precipitate that dissolves in both $\text{NH}_3(\text{aq})$ and H^+ indicates CO_3^{2-} .

A precipitate that dissolves in $\text{NH}_3(\text{aq})$ but not in H^+ indicates Cl^- .

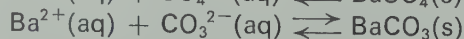
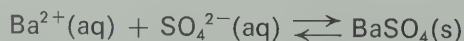
3. A precipitate with both Ba^{2+} and Ag^+ indicates CO_3^{2-} .

K_{sp} values:

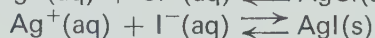
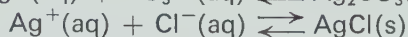
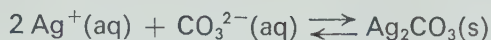


PROCESSING THE DATA

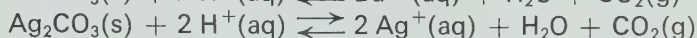
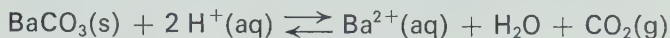
- 1) Write net ionic equations for the precipitation reactions which occurred when solutions of the anions were mixed with the reagent containing Ba^{2+} ions.



- 2) Do the same for precipitation reactions which occurred in Step c with the Ag^+ ions.



- 3) Write equations for the reactions which occurred when the precipitates were acidified with 1.0 M HNO_3 .



- 4) Write equations for the reactions which occurred when the silver precipitate reacted with aqueous ammonia to form the complex ion, $\text{Ag}(\text{NH}_3)_2^+$.



Experiment 24

A Study of Chemical Reactions

PURPOSE AND SUMMARY

The student performs a great variety of simple and, in many cases unrelated, tests. He gains experience observing the more obvious changes that take place during chemical reactions.

TIMING

Anytime during Chapter 9 is suitable. Do Parts I and II one day (40 minutes) and Parts III and IV another day (30 minutes).

MATERIALS NEEDED

per student

ring stand, ring, wire gauze, and burner

6 or more 13×100 mm test tubes (2 must be hard glass. See Lab Hint 1.)

1 beaker, 100 ml

1 beaker, 250 ml

thermometer, -20° to 150°C

wood splint

plastic square thumb protectors

solid reagents, in small bottles. See Lab Hint 2. Roman number after chemical indicates part of experiment in which it is needed.

ammonium chloride, 1 ml ($\frac{2}{3}$ g), I

calcium carbonate (marble chips) 5 small chips, III

lead dioxide, powdered, $\frac{1}{2}$ ml ($\frac{1}{2}$ g), II

sodium acetate, 1 ml ($\frac{2}{3}$ g), I
sodium hydroxide pellets, 3 pellets ($\frac{1}{3}$ g), I
sodium sulfite (or sodium bisulfite) $\frac{1}{4}$ ml ($\frac{1}{4}$ g), II

solutions. See Lab Hint 2.

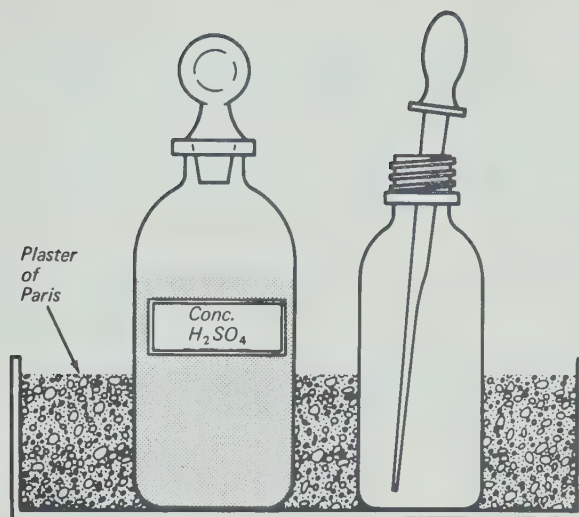
acetic acid, 5 ml 6 *M* (340 ml conc./liter) III
acetic acid, 5 ml 1 *M* (57 ml conc./liter) III
ferrous sulfate, acidified, 5 ml 0.1 *M* (28 g $\text{FeSO}_4 \cdot 7 \text{H}_2\text{O}$ /liter plus a few drops of H_2SO_4 , freshly prepared) II
hydrochloric acid, 8 ml 6 *M* (513 ml conc./liter) II, III
hydrochloric acid, 5 ml 1 *M* (85.5 ml conc./liter) III
hydrochloric acid, 5 ml 0.1 *M* (100 ml 1 *M* HCl/liter) III
manganous sulfate, $\frac{1}{2}$ ml 0.1 *M* (17 g $\text{MnSO}_4 \cdot \text{H}_2\text{O}$ /liter) III
phenolphthalein (1 g/100 ml 50% ethanol) II
potassium bromide, 2 ml 0.1 *M* (12 g KBr/liter) II
potassium chromate, 2 ml 0.1 *M* (19 g K_2CrO_4 /liter) IV
potassium permanganate, 1 ml 0.1 *M* (16 g KMnO_4 /liter) II, III
silver nitrate, 2 ml 0.2 *M* (34 g AgNO_3 /liter) IV
sodium chloride, 4 ml 0.1 *M* (6 g NaCl/liter) II, III
sodium oxalate, 20 ml 0.1 *M* (13 g $\text{Na}_2\text{C}_2\text{O}_4$ /liter) III
sulfuric acid, 1 ml 18 *M*. See Lab Hint 3. I
sulfuric acid, 1 ml 2.0 *M* (111 ml conc./liter) III

per class

Several bottles of saturated sodium hydrogen carbonate should be available for neutralizing spilled acid.

LAB HINTS

1. The test tube used in Part II j will be stained and difficult for the students to clean. It is therefore suggested that the same test tubes be used for all your classes and then saved for next year.
2. Make as many sets of reagents available as possible in order not to waste time. Since the order is not important, different students may begin on different parts.
3. Dispensing drops of conc. H_2SO_4 poses several problems. The container should be resistant to the acid, and acid should not easily run down the outside. All glass dropper bottles (Fischer 3-000, 125 ml) require constant attention to the tapered stopper. Plastic bottles tend to leak. One method is to use a set consisting of a reagent bottle (125 ml or smaller) with glass stopper and a small bottle in which is stored a medicine dropper with a long glass tube. Place these in a wood block in which holes have been drilled or in a plastic container with plaster of Paris poured to make compartments for the bottles as in the diagram below. The student lifts the glass stopper in the usual way, dips the dropper into the reagent bottle, dispenses the required number of drops of acid, empties the dropper, and replaces it in the small bottle and the stopper in the reagent bottle. The same medicine dropper is used by all students, and since there is no rinsing, the acid does not become diluted, and contamination is minimized. It is, of course, important to explain that this is contrary to the usual instruction never to put any object into a reagent bottle and never to return unused portions to the bottle. But it gives you a chance to make a very special case of a situation that is potentially dangerous both to the student's person and to his clothing.



PRELAB DISCUSSION

Tell the student there are two purposes to this experiment: to look for evidence of chemical reaction and to gain experience with a set of reactions that will be used later. All students have observed some chemical reactions, either at home or in other science classes, but it is convenient to have a set of reactions which have been seen under similar conditions by each student.

Do not assign or discuss the equations at this time. These reactions involve principles of oxidation-reduction, acid-base equilibria, reaction rates, and solubility, which are important topics in the next few chapters. The student is to observe now; the details will come later.

Urge students to use a marked tube for Part II j. They are very difficult to clean. Graduates are not needed. Volumes can be estimated satisfactorily. Good notes are essential if this experiment is to be useful to later chapters. In Part III k, do not wait for reactions. Have students put the tubes aside and glance at them occasionally as they go ahead to other parts.

PRECAUTIONS

Special precautions are needed because of the conc. H₂SO₄. Caution of the danger of adding water to this acid. Acid spills should be washed with an abundance of water. Sodium bicarbonate solution should be used to neutralize any acid that remains after washing. Wear safety glasses and use protective aprons.

POSTLAB DISCUSSION

Summarize the kinds of changes that have been observed—energy changes, color changes, production of substances with different properties. You may wish to emphasize the importance of using several different senses in noting changes in temperature, odor, and appearance. The presence of factors affecting the rate of a reaction may be briefly mentioned, but Chapter 12 is the place for full treatment. Do not discuss the reactions. The following is for your information only.

SAMPLE DATA

PART I

What the student observes is the sum of two processes. In the concentrated H_2SO_4 and in the crystal lattice of NH_4Cl , NaCH_3COO , and NaOH , the particles attract each other, and considerable energy is required to break these bonds. A solvent such as water combines with the ions and releases energy. The heat of hydration of some substances (for example, NaCl) just about offsets the heat of separation of the ions, and there is very little heat effect. For sulfuric acid and NaOH the heat of hydration more than offsets the energy required to separate the ions, and heat is evolved.

With NH_4Cl and NaCH_3COO more energy is required to break up the crystal lattice than is produced from the hydration of the ions, and heat is absorbed. When one mole of NH_4Cl is dissolved in 200 moles of water, ΔH is $+3.9$ kcal. The student may not observe this, however, because of the poor heat conductivity of the glass test tube. Direct him to check the temperature change with a thermometer. A similar amount of energy is absorbed when NaCH_3COO dissolves.

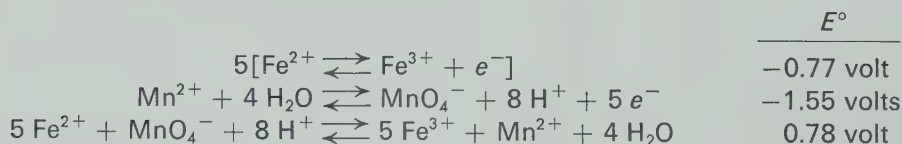
PART II

- e) and f) Phenolphthalein changes from colorless to pink between pH 8 and 9, hence it is colorless in the H_2SO_4 solution and pink in the sodium hydroxide solution.
g) The odor of SO_2 will be noted.



The student may also note the odor of the HCl , although he may not recognize either. Encourage him to relate them to other, more familiar odors.

- h) The pink color of the MnO_4^- disappears, and the yellow typical of Fe^{3+} appears.

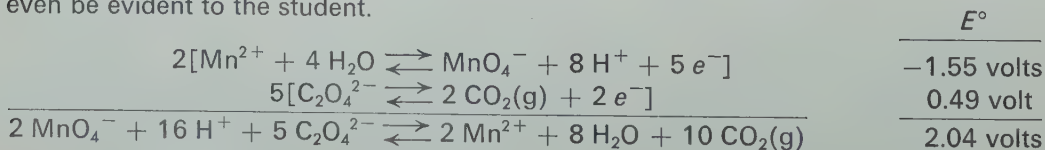


- i) No change in properties is noted. The resulting solution is a mixture of hydrated ions.
j) $\text{PbO}_2(\text{s}) \longrightarrow \text{PbO}(\text{s}) + \frac{1}{2} \text{O}_2(\text{g})$

The PbO_2 decomposes at 290°C , and the PbO melts at 888°C . The test tube is generally heated enough to fuse some of the PbO with the glass. The student may have some difficulty in getting a glowing splint to burst into flame, but having seen the film "Gases and How They Combine" for Chapter 2, he will probably succeed after a few trials.

PART III

- k) The tube to which Mn^{2+} is added shows a reaction—evidenced by bleaching of the MnO_4^- —much sooner than the one without Mn^{2+} . The reaction at 45°C is more rapid than that at room temperature. In fact it is very slow at room temperature without Mn^{2+} and may not even be evident to the student.

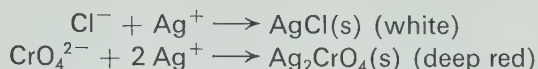


At these concentrations CO_2 gas will not be noticed. If the water is very alkaline, there may be a brown precipitate of MnO_2 rather than colorless Mn^{2+} .

- l) The evolution of gas (CO_2) is clearly more rapid with 6 *M* HCl than with any of the other acids. The 6 *M* acetic acid will react like the 0.1 *M* HCl, and the 1 *M* acetic acid will show very little bubbling.

PART IV

m)–o)



When NaCl and K_2CrO_4 are mixed, the addition of Ag^+ first forms a white precipitate which, because of the yellow CrO_4^{2-} solution, appears creamy yellow. After about ten drops ($\frac{1}{2}$ ml) have been added, the deep red characteristic of Ag_2CrO_4 develops and masks the white AgCl.

The K_{sp} for AgCl is 1.7×10^{-10} and for Ag_2CrO_4 is 1.9×10^{-12} at 25°C.

When both precipitates are present, there will be only one concentration of Ag^+ , and both K_{sp} values will be satisfied.

$$[\text{Ag}^+][\text{Cl}^-] = 10^{-10} \quad \text{and} \quad [\text{Ag}^+]^2[\text{Cl}^-]^2 = 10^{-20}$$

Solving $[\text{Ag}^+]^2[\text{CrO}_4^{2-}] = 10^{-12}$ for $[\text{Ag}^+]^2$ and substituting gives

$$\frac{[\text{Cl}^-]^2}{[\text{CrO}_4^{2-}]} = 10^{-8}$$

If the concentration of CrO_4^{2-} is 0.04 *M*, as it will be when diluted to 2.5 ml in this procedure, then

$$[\text{Cl}^-]^2 = 0.04 \times 10^{-8} = 4 \times 10^{-10}$$

and

$$[\text{Cl}^-] \cong 2 \times 10^{-5} \text{ mole/liter}$$

As long as the $[\text{Cl}^-]$ exceeds 2×10^{-5} mole/liter, the $[\text{Ag}^+]$ will not be great enough for $[\text{Ag}^+]^2[\text{CrO}_4^{2-}] = 10^{-12}$, and Ag_2CrO_4 will not precipitate.

RESULTS OF PROCESSING THE DATA

- 1) In which of the experiments was there no evidence of a chemical reaction?

Parts II k (phenolphthalein and sulfuric acid), II i (NaCl and KBr solutions), and possibly the 1 *M* acetic acid in III l show little evidence for chemical reaction. The heat effects in Parts I a–d might be sufficient to classify these as chemical reactions. The hydrated ions are certainly not the same species as the unhydrated ions.

- 2) Which chemical reactions produced a new phase?

Gases were produced in Parts II g, j, and III l. Precipitates were found in IV m–o.

- 3) Which reactions in this experiment were exothermic? Which were endothermic?

Evolution of heat was noted in Parts I a and I b, in the demonstration, and possibly in II e and III l. Endothermic reactions were observed in Parts I c, I d, and II j.

- 4) In which reactions did an increase in temperature affect the rate?

An increase in temperature increased the rate in Part III k. The effect may also be noted for Parts II j and III l.

- 5) In which reactions did an increase in concentration affect the rate?

This was clearly evident in Part III l.

- 6) In Part III k what effect on the rate of reaction did adding the MnSO_4 solution have?

The rate was increased when MnSO_4 solution was added.

- 7) What evidence did you observe to indicate that in some of the reactions part of the reactants was not used up?

In Parts IV n and o, a precipitate continued to form until an excess of AgNO_3 solution was added, indicating that, until then, the reactant had not been used up. In Part III l, CaCO_3 was left after the bubbling ceased. In the experiments for which there is no evidence of reaction, the "reactants" might be considered unused.

In Part II h, several drops of KMnO_4 were decolorized. There must have been an excess of another reagent.

A QUESTION TO WONDER ABOUT

Account for the results obtained when solutions of hydrochloric acid and of acetic acid—of the same concentration—reacted with $\text{CaCO}_3(\text{s})$.

The student can think about this until Chapter 14.

DEMONSTRATION 27

AN ENDOTHERMIC REACTION

PURPOSE

To show a distinctively endothermic reaction. $\text{Pb}(\text{NO}_3)_2$ is heated in a hard glass test tube.

TIMING

About 10 minutes are needed during the *Postlab Discussion* for Experiment 24.

MATERIALS NEEDED

1 test tube, hard glass, 18×150 mm
 $\text{Pb}(\text{NO}_3)_2(\text{s})$, about $\frac{1}{2}$ ml
burner

LAB HINT

The test tube is difficult to clean. Save it for this demonstration.

CLASSROOM PRESENTATION

Heat the tube with the $\text{Pb}(\text{NO}_3)_2$ in the bottom. Amid popping and crackling, a brown gas forms. This gas is very TOXIC. Avoid breathing fumes. Remove tube from flame; noise and evolution of gas ceases. Heat is needed to make the decomposition take place. The reaction is endothermic. An equation describing the reaction is:



Chemical Bonding



The major types of chemical bonds are discussed and shown to have a common cause: the simultaneous attraction of electrons to more than one positive nucleus. This material is more conceptual than experimental in nature. The single experiment is really an introduction to the heat effects to be studied in Chapter 11.

Schedule of Experimental Work

TEXT SECTION	EXPERIMENTAL WORK	FOR DETAILS SEE PAGE
10-1 to 10-8	Expt. 25 (Any time)	128

Experiment 25

Energy of Combustion

PURPOSE AND SUMMARY

The student collects in a can of water, a good fraction of the heat produced when a candle burns. Knowing the grams of wax burned, the grams of water heated, and the change in temperature of the water, he calculates the heat of combustion per gram of wax.

TIMING

During Section 10-4; 40 minutes are needed.

MATERIALS NEEDED

per student

candle mounted on a lid
cans. See Lab Hint 1.
6 mm glass rod, 15–20 cm long
2 or 3 ice cubes. See Lab Hint 2.
ring stand, ring
thermometer, -20° to 150°C

per class

balance, centigram. One per two students is adequate.
graduated cylinder, 500 ml. One per four students is adequate.

LAB HINTS

1. Have students bring cans from home. Two are needed for each set. One set will handle many classes and will last for years.

The "calorimeter" can may be size 300, 303, or a small soup can. See Lab Manual Figure 25-1. Punch holes in the soup cans before class in order to save time and to avoid accidents. Use an ice pick to start the hole, and enlarge it with the end of a file.

The chimney should be a large juice can (size 3). Instruct the students to punch holes in the can with an old fashioned beer can opener before removing the bottom of the can. An alternate method for obtaining a draft through the "chimney" is to elevate the can by placing it on three rubber stoppers.

2. If the cold water from the tap is 10 or 15°C below room temperature, ice will not be needed in procedure (e). Starting below and finishing above room temperature tends to equalize heat exchange with the room.

PRELAB DISCUSSION

Little discussion should be needed. Encourage students to use the same balance for all weighings, and caution them about loss of wax in the form of drippings. The can should be placed directly over the candle as soon as possible after lighting the candle. Have the students record their values for cal/g on a wall chart. Such a compilation gives a basis for discussing both the accuracy and the precision involved in this experiment.

PRECAUTIONS

DO NOT get hair too close to flame. Take care that soot does not get on clothing. Wear an apron.

POSTLAB DISCUSSION

Several points can be made while discussing the class results of this experiment. The uncertainty calculations are not very difficult. They can be used to give more meaning to the $\pm$ notation. The range indicated by the $\pm$ usually includes the majority of values obtained.

Heat of combustion is as characteristic of a substance as is boiling temperature. This can be developed further if some students wish to extend the experiment by using an alcohol lamp as a source of heat. Ethyl alcohol has a heat of combustion of 7.1 kcal/g and methyl alcohol, 5.3 kcal/g.

SAMPLE DATA AND RESULTS OF PROCESSING THE DATA**1) MASS OF CANDLE BURNED**

Mass of candle before 32.65 ± 0.01 g

Mass of candle after 31.23 ± 0.01 g

Mass of candle burned = 1.42 ± 0.02 g or $1.42 \pm 1.4\%$

2) MASS OF WATER HEATED

Volume of water heated 321 ± 2 ml

(assuming the graduated cylinder can be read ± 2 ml).

Mass of water heated 321 ± 2 ml $\times \frac{1 \text{ g H}_2\text{O}}{1 \text{ ml H}_2\text{O}} = 321 \pm 2$ grams

3) TEMPERATURE CHANGE OF WATER, Δt

Temperature of water after $40.2 \pm 0.2^\circ\text{C}$

Temperature of water before $10.2 \pm 0.2^\circ\text{C}$

$\Delta t = 30.0 \pm 0.4^\circ\text{C}$

4) HEAT REQUIRED TO WARM THE WATER

This is given by (mass of water)(Δt) $\left(\frac{1 \text{ cal}}{\text{g}(^\circ\text{C})} \right)$

$(321 \pm 2 \text{ g})(30.0 \pm 0.4^\circ\text{C})(1)$

or

$(321 \pm 0.6\%)(30.0 \pm 1.3\%)(1)$
 $9630 \pm 1.3\%$ or 9630 ± 120

5) HEAT OF COMBUSTION PER GRAM OF WAX

This is given by $\frac{\text{quantity of heat from combustion}}{\text{mass of candle burned}}$

We obtain

$$\frac{9630 \pm 1.3\% \text{ cal}}{1.42 \pm 1.4\% \text{ g}} = (6800 \pm 1.4\%) \text{ cal/g}$$

$$= 6800 \pm 95 \text{ cal/g}$$

If only one uncertain digit is to be shown, this should be reported as $6800 \pm 100 \text{ cal/g}$. This is a median student answer.

- 6) Do you think an experiment using a more refined calorimeter would give a higher or a lower value than the one you determined in Question 5? Explain.

Since we assumed all the heat from the burning candle was used to heat only the water, when actually some was used to heat the cans and some was lost to the air, the calculated heat of combustion should be lower than a more rigorously determined value. In the

example above, the fraction might be $\frac{14,000}{1.42}$ to give a better value of 9900 cal/g . The

handbook value is about $10,300 \text{ cal/g}$. This varies a bit depending on the type of wax used.

- 7) Most candles are made of a mixture of waxes. Assume yours was a pure wax with the formula $\text{C}_{25}\text{H}_{52}$. Calculate the heat of combustion of $\text{C}_{25}\text{H}_{52}$, $\Delta H_{\text{combustion}}$, in kcal/mole.

The molar mass of $\text{C}_{25}\text{H}_{52}$ is $25(12) + 52(1) = 352 \text{ g/mole}$

$$\begin{aligned}\Delta H_{\text{combustion}} &= (352 \text{ g/mole})(6800 \pm 100 \text{ cal/g}) \\ &= (2,400,000 \pm 1.4\%) \text{ cal/mole} \\ &= 2,400,000 \pm 34,000 \text{ cal/mole} \\ &= 2400 \pm 34 \text{ kcal/mole or better, } 2400 \pm 30 \frac{\text{kcal}}{\text{mole}}\end{aligned}$$

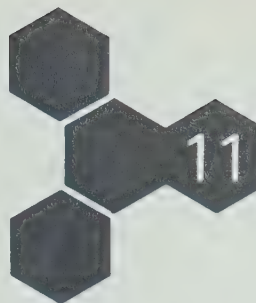
The uncertainty in the molar mass is small enough to be ignored.

- 8) (Optional) Assume some black soot formed on the bottom of the can of water during your experiment. Would this contribute to a high or a low value for $\Delta H_{\text{combustion}}$? Explain.

The soot represents unburned carbon. Had it burned, more heat would have been available. The numerator of the fraction $\frac{\text{calories of heat absorbed by water}}{\text{grams wax burned}}$ is smaller than

it would have been had the soot burned. Formation of soot contributes to a low value for $\Delta H_{\text{combustion}}$. Some might argue that the black soot makes a better heat absorber than the bright can. It does. But so would a very thin film. A thick mass of soot acts as an insulator. A very thin deposit of soot might contribute to a higher than class average value for $\Delta H_{\text{combustion}}$.

Energy Effects in Chemical Reactions



Energy has a part of every reaction. This chapter makes that point and, through the experiment, the student learns of the additive quality of the heat produced or consumed. The heat effect will be connected to the activated complex theory of reaction rates in the next chapter.

Schedule of Experimental Work

TEXT SECTION	EXPERIMENTAL WORK	FOR DETAILS SEE PAGE
11-1 to 11-3		
	Expt. 26	132
11-4		
	Expt. 27*	135
11-5 to 11-7		

* Placement not critical; should be after Expt. 26.

Experiment 26

Heat of Reaction

PURPOSE AND SUMMARY

To give the student an opportunity to discover experimental evidence for the Law of Additivity of Heat.

The student measures and compares the quantity of heat energy released in three exothermic reactions. He discovers that one of the reactions is the sum of the other two and that the heats of reaction are related in the same way the equations are related. The equations for the three reactions are:

1. $\text{NaOH(s)} \longrightarrow \text{Na}^+(\text{aq}) + \text{OH}^-(\text{aq}) + x_1 \text{ kcal}$
2. $\text{NaOH(s)} + \text{H}^+(\text{aq}) + \text{Cl}^-(\text{aq}) \longrightarrow \text{H}_2\text{O} + \text{Na}^+(\text{aq}) + \text{Cl}^-(\text{aq}) + x_2 \text{ kcal}$
3. $\text{Na}^+(\text{aq}) + \text{OH}^-(\text{aq}) + \text{H}^+(\text{aq}) + \text{Cl}^-(\text{aq}) \longrightarrow \text{H}_2\text{O} + \text{Na}^+(\text{aq}) + \text{Cl}^-(\text{aq}) + x_3 \text{ kcal}$

TIMING

Since the topic is discussed there, this experiment should be done before Section 11-4 is assigned.

One 45–50 minute period is adequate.

MATERIALS NEEDED

per student or pair

- 1 Styrofoam cup, 8 oz
- 1 beaker, 250 ml
- 1 thermometer, -20° to 150°C
- 1 balance, centigram
- 4 grams of solid NaOH. See Lab Hint 1.
- 100 ml 0.5 *M* NaOH (20 g/liter)
- 100 ml 0.5 *M* HCl (42.8 ml conc. reagent/liter)
- 200 ml 0.25 *M* HCl (21.4 ml conc. reagent/liter)
- 200 ml distilled water. See Lab Hint 2.

for class

- 1 dispensing bottle for each solution and distilled water
- 1 small reagent bottle of NaOH pellets for each balance
- 1 graduated cylinder or volumetric flask, 100 ml, for each solution. See Lab Hint 3.
- 1 squeeze bottle of vinegar or 0.1 *M* acetic acid. See Precautions.

LAB HINTS

1. Sodium hydroxide pellets appear to pick up quite a bit of moisture when exposed to air, but experience indicates that only a small increase in mass occurs during the time it takes to weigh the sample. See the table on page 133.

Mass Gained by NaOH

(air temperature, 29°C, and relative humidity about 35%)

Time, minutes	Mass of sample, grams
0	2.00
5	2.01
10	2.02
20	2.03
30	2.04
40	2.05
50	2.06
60	2.07

Apparently the amount of water is more noticeable than measurable ! It is important, however, to tell the student approximately how many pellets to use and also to have the NaOH in small reagent bottles by each balance in order that the bottles can be closed promptly. Each student will probably need between 10 and 20 pellets. This should be determined in advance.

2. Solutions must be made up at least one day before they are to be used and stored at the dispensing location so that they will all be at room temperature.
3. To avoid the problem of dilution and contamination of solutions, have a 100 ml graduated cylinder or volumetric flask available at each dispensing bottle. Clearly label each cylinder or flask and attach them to the dispensing bottle with a piece of string.
4. You may wish to discuss whether the number of calories is found from the change in temperature multiplied by just the mass of water or multiplied by the total mass of water, NaOH, and HCl. The increase in mass is only about 2%, and some of this is counterbalanced by the fact the specific heat of the solution is slightly less than for pure water. An error of 2% is small compared to the precision of the other measurements ; so it may be neglected.
5. You may wish to have the students save the 0.25 *M* NaOH solution prepared in step c. Have a labeled container available for this purpose but placed where it will not be confused with the prepared solutions for *Reactions* 2 and 3.

PRELAB DISCUSSION

The students have already used a simple calorimeter in Experiments 13, 14, and 25. There is little difference in the one used in this experiment. Using the Styrofoam cup, we can neglect the heat used to warm the cup. If some of the students become interested in improving the accuracy of the experiment, they can use a control system to determine the heat loss due to the interaction with the air and the cup. Or some of the students may want to determine the water equivalent for the Styrofoam calorimeter using one of the reactions as the standard. See page 203 of the Teacher's Guide for further discussion of this technique.

Make it clear that reactants should be mixed together as quickly as possible to minimize heat loss to the environment. Remind the students to read their thermometers as precisely as possible.

Point out that they should use as close to 2.00 grams of NaOH(s) as possible, but they should not waste time being too precise. See Lab Hint 2. Perhaps you would like to suggest they check the resulting solutions in each reaction using litmus paper.

PRECAUTIONS

Discuss the dangers and the problems involved in using solid sodium hydroxide. Students should not touch it with their fingers, and if some is spilled it should be picked up **immediately** using a paper towel and discarded in a waste jar or dissolved in the sink. If any is handled accidentally, have them wash their hands thoroughly and if slipperiness persists use a little vinegar or dilute (0.1 *M*) acetic acid (have a jar or squeeze bottle available).

POSTLAB DISCUSSION

This experiment leads so directly into the material of Section 11-4 that you should combine discussion of the experiment with a discussion of the Textbook assignment. Tabulate class data to emphasize the regularities observed. Individual students may obtain unsatisfactory data, but the class data should be convincing.

RESULTS OF PROCESSING THE DATA

- 1) Prepare a table such as the one shown and make the necessary calculations in order to complete it.

<i>Reaction</i>	Δt C°	Heat Energy Released cal	Moles of NaOH Used	Energy Released per Mole NaOH cal
1	2.6	520	0.052	1.0×10^4
2	5.8	1200	0.048	2.5×10^4
3	3.4	680	0.050	1.4×10^4

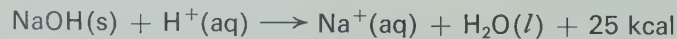
- 2) Use the ΔH notation to express the energy released per mole of NaOH for each reaction: ΔH_1 , ΔH_2 , and ΔH_3 . Use two significant figures in all calculations.

$$\Delta H_1 = -10 \text{ kcal/mole}$$

$$\Delta H_2 = -25 \text{ kcal/mole}$$

$$\Delta H_3 = -14 \text{ kcal/mole}$$

- 3) Write net ionic equations for *Reactions* 2 and 3.



- 4) ΔH_1 , in *Reaction* 1, represents the energy of solution for one mole of NaOH(s). Look at the net ionic equations for *Reactions* 2 and 3, and make a similar statement concerning the significance of ΔH_2 and ΔH_3 .

ΔH_3 represents the heat of reaction for one mole of $\text{H}^+(\text{aq})$ and one mole of $\text{OH}^-(\text{aq})$.

ΔH_2 represents the sum of ΔH_1 and ΔH_3 ; the heat of solution of NaOH(s) plus the heat of reaction of $\text{H}^+(\text{aq})$ and $\text{OH}^-(\text{aq})$. *Reaction 1 + Reaction 3 = Reaction 2.*

- 5) Compare ΔH_2 with $(\Delta H_1 + \Delta H_3)$. Account for any similarity or difference.

$$\Delta H_2 = -25 \text{ kcal/mole}$$

$$\Delta H_1 + \Delta H_3 = (-10 \text{ kcal/mole}) + (-14 \text{ kcal/mole})$$

$$= -24 \text{ kcal/mole}$$

$$\Delta H_2 - (\Delta H_1 + \Delta H_3) = -1 \text{ kcal/mole}$$

As indicated in 4), *Reaction 2* is the sum of *Reactions 1* and 3, therefore we would expect the ΔH 's for the reactions to compare in the same way. Any differences are probably due to experimental error and uncertainty.

- 6) Calculate the percent difference between ΔH_2 and the sum ($\Delta H_1 + \Delta H_3$). Assume ΔH_2 to be correct.

$$\frac{-1 \text{ kcal/mole}}{-25 \text{ kcal/mole}} \times 100 = 4\%$$

This is well within the limits of uncertainty in the measurements in this experiment. The difference is not significant.

- 7) Suppose you had used 4.00 grams of NaOH(s) in *Reaction 1*. What would have been the number of calories released in the reaction? What effect would this have on your calculation of ΔH_1 ?

The total number of calories released would be twice as much as for 2.00 grams. No effect on ΔH_1 . Twice the amount of heat would be divided by twice the number of moles of NaOH(s), thus, the value of ΔH_1 would be about the same. The degree of precision would be greater since the least precise measurement involves temperature. In *Reaction 1* the degree of uncertainty for Δt is about 10%!

Experiment 27

Heat of Reaction for the Combustion of Magnesium

PURPOSE AND SUMMARY

To give the student additional experience in the application of the Heat Additivity Law, and to illustrate how the heat term can be obtained for a reaction that is inconvenient to perform.

The equations for three reactions are given which can be combined to give a fourth equation. The sum of the three equations represents the oxidation of magnesium. The heat of reaction is determined calorimetrically for two of the reactions and the third is obtained from the table in the Textbook.

TIMING

This experiment should be done during Chapter 11 and following Experiment 26 so that the student first obtains evidence that ΔH 's are additive and then in this experiment he applies the regularity.

A period of about 30 minutes is needed. There will probably be time for some of the students to repeat the experiment during a full period.

MATERIALS NEEDED**per student or pair**

- 1 balance, centigram
- 1 graduated cylinder, 100 ml
- HCl solution 200 ml, 1.00 *M*, (85.6 ml conc. reagent/liter). See Lab Hint 1.
- magnesium oxide, 1 gram. See Lab Hint 2.
- magnesium ribbon, 0.5 gram. See Lab Hint 3.
- 1 Styrofoam cup, 8 oz
- 1 thermometer, -20° to 150°C

LAB HINTS

1. Make the 1.00 *M* HCl solution available in dispensing bottles. Prepare the solution at least one day before it is to be used so that the temperature will be at room temperature.
2. Have reagent bottles of MgO placed near each balance.
3. Cut pieces of shiny, bright magnesium ribbon (free of corrosion) into pieces that are about 0.5 gram. Make these pieces available near each balance.

PRELAB DISCUSSION

If any of your students have difficulty combining the equations (2, 3, and 4) to obtain equation (1), help them do this before they do the experiment. There is little need for further discussion.

PRECAUTIONS

Observe the usual lab precautions. Have the students wear safety glasses and aprons.

POSTLAB DISCUSSION

Discuss the calculations in *Processing the Data*. Make a histogram of the class data for ΔH_1 . Have selected students compare the best class value to handbook information.

SAMPLE DATA AND CALCULATIONS

Magnesium and HCl
mass of Mg used to react
with 100 ml 1 *M* HCl

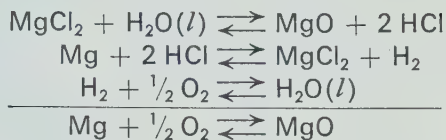
initial temperature	24.8°C
final temperature	49.0°C
Δt	24.2 C°
kcal energy released	2.42
moles Mg used	0.0226
kcal/mole Mg	107

Magnesium Oxide and HCl
mass of MgO used to react
with 100 ml 1 *M* HCl

initial temperature	25.2°C
final temperature	29.2°C
Δt	4.0 C°
kcal energy released	0.40
moles MgO	0.012
kcal/mole MgO	33

RESULTS OF PROCESSING THE DATA

- 1) Combine equations (2), (3), and (4) to obtain equation (1).



- 2) Calculate the change in temperature, Δt , for each reaction, (2) and (3).

$$\begin{aligned}
 \Delta t &= \text{final temperature} - \text{initial temperature} \\
 &= 29.2 - 25.2 = 4.0 \text{ C}^\circ \quad \text{for } \text{MgO} + \text{HCl} \text{ reaction} \\
 &= 49.0 - 24.8 = 24.2 \text{ C}^\circ \quad \text{for } \text{Mg} + \text{HCl} \text{ reaction}
 \end{aligned}$$

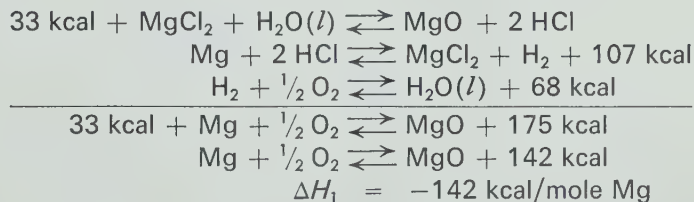
- 3) Calculate the kcal of energy released for each reaction. (Assume that one calorie of energy is required to change the temperature of one ml of solution one degree Celsius.)

$$\begin{aligned}
 \text{kcal released} &= \Delta t \times 1 \text{ cal/ml(C}^\circ) \times \text{ml solution} \\
 4.0 \text{ C}^\circ \times 1 \text{ cal/ml(C}^\circ) \times 100 \text{ ml} &= 400 \text{ cal} = 0.40 \text{ kcal (MgO reaction)} \\
 24.2 \times 1 \times 100 &= 2420 \text{ cal} = 2.42 \text{ kcal (Mg reaction)}
 \end{aligned}$$

- 4) Calculate kcal/mole Mg and kcal/mole MgO.

$$\begin{aligned}
 2.42 \text{ kcal}/0.0226 \text{ mole Mg} &= 107 \text{ kcal/mole Mg} \\
 0.40 \text{ kcal}/0.012 \text{ mole MgO} &= 33 \text{ kcal/mole MgO}
 \end{aligned}$$

- 5) Determine the ΔH for reaction (1). Report ΔH in kcal/mole of MgO.



The Rates of Chemical Reactions



The experimental measurement of a reaction rate is done in Experiment 28. The Textbook chapter concentrates on the concept of the activated complex. That theory is shown to coordinate the effects of temperature and concentration changes. Catalytic behavior is also covered. Details of the activated complex are difficult to determine, even for experienced research workers. Such experiments are clearly beyond the high school student.

Schedule of Experimental Work

TEXT SECTION	EXPERIMENTAL WORK	FOR DETAILS SEE PAGE
12-1	Demo. 28	139
12-2 to 12-9	Expt. 28	140

DEMONSTRATION 28

A CHANGE IN REACTION RATE

PURPOSE

To show a dramatic change of rate in a combustion reaction.

TIMING

About 5 minutes are needed during an introduction to Chapter 12.

MATERIALS NEEDED

A one-gallon, cylindrical paint can. The lid must have almost the same diameter as the can, and be the type that is pressed to fit. Punch a 0.5 cm hole in the center of the lid, and a 1 cm hole near the bottom of a vertical, curved side.

A source of lab gas. Propane gas (from a bottle) is unsuitable. It burns too fast. An explosion occurs as soon as it is ignited.

CLASSROOM PRESENTATION

Put the lid on snugly. The tighter it fits, the higher it will blow! Use a rubber hose to fill the can with lab gas *via* the hole in the lid. Be sure the can is full of gas and that most of the air is driven out. Hold your fingers over each hole and move the can to a safe place. The lid should not blow up to hit a light fixture. Nor should it be accessible to students while it burns. Light the gas at the hole in the lid.

All of these operations can be accompanied by a patter about a convenient tent warmer you have invented. Warm your hands over the flame in an appreciative manner. Move away and continue to talk about different kinds of reactions, and how they differ. Some are slow; some are fast. To the inevitable question, "How long will the heater burn," answer "Until it goes out," or point out an experiment is being done right now to determine the answer to that question. About 3 minutes after being lit, the gas in the can will explode. This is the time to emphasize that the rate of a chemical reaction is a very important characteristic of the reaction.

Much of the explanation for this behavior can be drawn out of the class. The gas *inside* the can did not burn initially because there was not enough air in the can to support combustion. As some gas burned, air entered the can *via* the bottom hole. The air and lab gas mixed until the concentrations were suitable for combustion inside the can. The combustion released heat into a combustible mixture which proceeded to react at a great rate. The effects of concentration and temperature on reaction rate are discussed in this chapter.

Experiment 28

A Study of Reaction Rates

PURPOSE AND SUMMARY

To determine quantitatively the influence of temperature, concentration, and a catalyst on the time required for a reaction to occur.

The students study the effect of changing the concentration of the reactants in Part I, the effect of changing the temperature in Part II, and the effect of adding a catalyst in Part III. Class data is graphed by the students in order to make the effects more visual and the generalizations more obvious.

TIMING

The experiment should be done before Section 12-2 in the Textbook. If all solutions are readily available, the students will be able to do all the assigned mixtures within a single class period. You may wish to break the experiment up into three shorter sessions so that each pair can do more of the mixtures. It is better, however, to collect all the data the same day.

MATERIALS NEEDED

per student or pair

- 3 graduated cylinders, 25 ml
- 1 beaker, 100 ml
- 1 thermometer, -20° to 150°C
- 1 stirring rod, glass
- 3 test tubes, 18×150 mm

per class

1 clock with sweep-second hand

Solutions. See Lab Hint 1.

Solution A. 0.20 *M* potassium iodide solution
(33.2 grams KI/liter of solution)
with 10 ml of 3% starch solution/liter KI solution

Solution B. 0.0050 *M* sodium thiosulfate solution
(1.24 grams $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5 \text{H}_2\text{O}$ /liter of solution)

Solution C. 0.10 *M* ammonium peroxydisulfate solution
(22.8 grams $(\text{NH}_4)_2\text{S}_2\text{O}_8$ /liter of solution)

Catalyst Solution. 0.01 *M* copper sulfate solution
(2.50 grams $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$ /liter of solution)

LAB HINTS

1. You will need about 2 liters of solutions *A* and *C* and about 1 liter of solution *B* per class of 30 students. One liter of the catalyst solution will be enough for about 5000 students. Prepare the solutions as indicated above and make them readily available to the students in plastic squeeze bottles. Add about 10 ml of 3% starch solution (3 grams soluble starch added to 100 ml boiling water) to each liter of the KI solution not far in advance of when you are to do the experiment. Make the 0.01 *M* copper sulfate solution available in dropper bottles.
2. Assign the students to work in pairs and tell them which mixtures they are to use. In Part I each pair should do *Mixture 1* and one other. In Parts II and III each pair should do at least one mixture.

3. Make arrangements in advance for the students to record their results so that class data may be shared by all. This can be done by listing the mixture numbers on the chalkboard and having the students record their results as they are obtained. Averages can be obtained for mixtures assigned to more than one pair.

PRELAB DISCUSSION

Discuss the introduction to the experiment with the students. Write the equations on the board and ask questions about the experiment in order to clarify their understanding of the reactions involved. Discuss the importance of making precise measurements. In order to obtain comparable data everyone must be careful to control the same variables. Remind the students that in the dilution mixtures they must add sufficient water to the solutions being diluted to give 20 ml samples each time. Solutions must not be accidentally diluted while being warmed or cooled.

The starch-iodine complex is discussed in *Scientific American*, July 1962, p. 88. An interesting problem can be posed by this quick demonstration. Stand a test tube containing the starch-iodine complex in a beaker of hot water ($\sim 70^\circ\text{C}$) so that only half the contents is heated. The color disappears from the bottom half. Why?

PRECAUTIONS

Observe the usual laboratory precautions.

POSTLAB DISCUSSION

Some of the students may need help with the calculations. Give them an opportunity to first work them out on their own giving them hints as needed. As you discuss their data with them and analyze their graphs, help them recognize the generalizations that are to be found in the data.

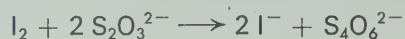
SAMPLE DATA AND RESULTS OF PROCESSING THE DATA

Part I. The Effect of Concentration

- 1) Prepare a table such as the one shown. Make the calculations needed to complete it.

Mixture	Volume of Sol. A 0.20 M KI (ml)	Moles of I^-	Volume of Sol. C 0.10 M $(\text{NH}_4)_2\text{S}_2\text{O}_8$ (ml)	Moles of $\text{S}_2\text{O}_8^{2-}$	Elapsed Time (sec)
1	20.0	4.0×10^{-3}	20.0	2.0×10^{-3}	36
2	15.0	3.0×10^{-3}	20.0	2.0×10^{-3}	50
3	10.0	2.0×10^{-3}	20.0	2.0×10^{-3}	86
4	5.0	1.0×10^{-3}	20.0	2.0×10^{-3}	178
5	20.0	4.0×10^{-3}	15.0	1.5×10^{-3}	55
6	20.0	4.0×10^{-3}	10.0	1.0×10^{-3}	87
7	20.0	4.0×10^{-3}	5.0	0.5×10^{-3}	199

- 2) In each of the mixtures you used 10.0 ml of *Solution B* (0.0050 M Na₂S₂O₃). Calculate, using equation (2), how many moles of I₂ will react with this much thiosulfate ion, S₂O₃²⁻. The elapsed time for each reaction is a measure of the time it takes to use up this much S₂O₃²⁻ and to produce *n* moles of I₂ according to equation (1).

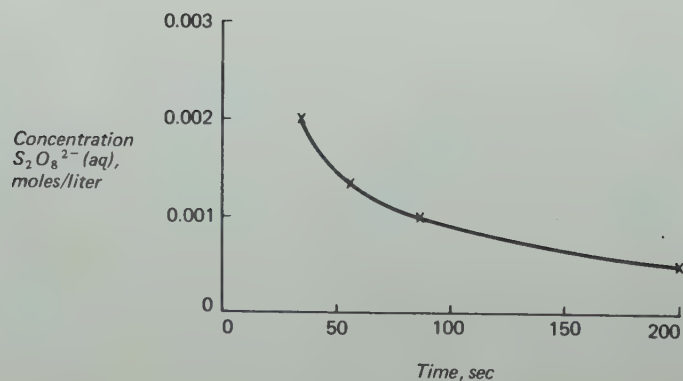
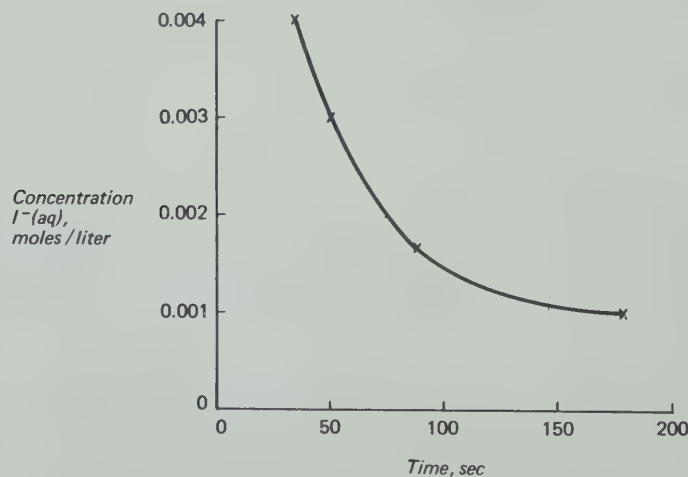


$$0.0050 \text{ M Na}_2\text{S}_2\text{O}_3 = \frac{0.0050 \text{ mole}}{1000 \text{ ml}} = \frac{5.0 \times 10^{-5} \text{ mole S}_2\text{O}_3^{2-}}{10 \text{ ml}}$$

$$5.0 \times 10^{-5} \text{ mole S}_2\text{O}_3^{2-} \times \frac{1 \text{ mole I}_2}{2 \text{ moles S}_2\text{O}_3^{2-}} = 2.5 \times 10^{-5} \text{ mole I}_2$$

- 3) Make a graph by plotting moles of iodide ion (*Solution A*) vs elapsed time — for *Mixtures 1–4*. Plot elapsed time for reactions along the horizontal axis.
- 4) Make another graph, plotting moles of peroxydisulfate ion (*Solution C*) vs elapsed time — for *Mixtures 1, 5, 6, and 7*. Plot elapsed time for reactions along the horizontal axis.
- 5) How does changing the concentration of reactants affect the elapsed time for the reaction?

The higher the concentration the shorter the elapsed time for the reaction. The lower the concentration the longer the time.



- 6) How many moles of $\text{S}_2\text{O}_8^{2-}$ are not used if all of the I^- in *Mixture 3* is completely changed to I_2 ?

$$2.0 \times 10^{-3} \text{ mole } \text{I}^- \times \frac{1 \text{ mole } \text{S}_2\text{O}_8^{2-}}{2 \text{ moles } \text{I}^-} = 1.0 \times 10^{-3} \text{ mole } \text{S}_2\text{O}_8^{2-}$$

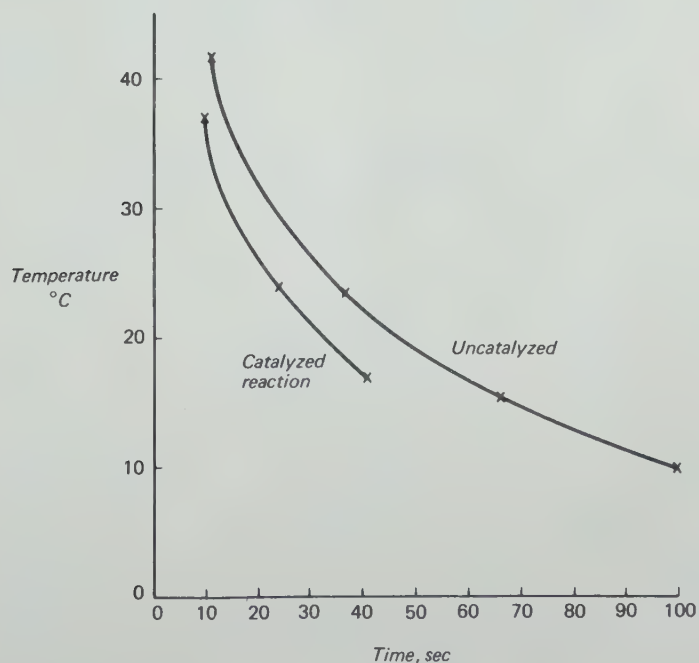
Since *Mixture 3* had 2.0×10^{-3} mole $\text{S}_2\text{O}_8^{2-}$ to start and 1.0×10^{-3} is needed to react with 2.0×10^{-3} mole I^- , 1×10^{-3} mole remains after all the I^- is changed to I_2 .

Part II. The Effect of Temperature

- 7) Prepare a table such as shown and make the calculations necessary in order to complete the table.

<i>Mixture</i>	Temperature (°C)	Elapsed Time (sec)
8	10.0	100
9	15.5	67
10	23.6	37
11	34.5	17
12	41.5	11

- 8) Make a graph plotting temperature of the mixture vs elapsed time for *Mixtures 8* through 12. Plot time along the horizontal axis.



- 9) What generalization can you deduce from these data?

As the temperature is increased the time for the reaction decreases.

10) How does a temperature change of about $10\text{ }^{\circ}\text{C}$ affect the elapsed time for the reaction?

At 20°C the time was 48 seconds.

At 30°C time was 23 seconds.

At 40°C time was 12 seconds.

Therefore, we can conclude a temperature change of about $10\text{ }^{\circ}\text{C}$ changes the elapsed time by a factor of 2. An increase of about $10\text{ }^{\circ}\text{C}$ cuts the time in half. This is only a crude rule of thumb. See Teacher's Guide, p. 227.

Part III. The Effect of a Catalyst

11) Prepare a graph plotting temperature of the mixture vs elapsed time, on the same graph prepared in Part II. Use the time-temperature data for *Mixtures* 13–17. Label this curve "catalyzed" and the other (from Part II) "uncatalyzed."

(See graph under question 8.)

12) What generalization can you deduce from a comparison of the two curves?

Adding a catalyst speeds up the rate reducing the elapsed time.

Equilibrium In Chemical Reactions



The equilibrium concept is a very important one. It is useful in the following chapters on acids and redox reactions. Also it is treated in more depth than has been usual in secondary chemistry classes. Experiment 29 offers a fine chance for the student to discover a regularity, especially with the guidance given by Demonstrations 29 and 30. The experiment leads to the constancy of the equilibrium law expression. Experiment 30 provides experience with the application of Le Chatelier's Principle.

Schedule of Experimental Work

TEXT SECTION	EXPERIMENTAL WORK	FOR DETAILS SEE PAGE
13-1, 13-2	Demo. 29	146
	Demo. 30	148
	Expt. 29	150
13-3 to 13-7	Expt. 30	171
	Demo. 31	175
13-8 to 13-15		
13-16		

DEMONSTRATION 29

CHEMICAL EQUILIBRIUM

PURPOSE AND SUMMARY

To introduce, in a qualitative way, the reaction for which the equilibrium constant is to be determined in Experiment 29. This reaction is also discussed in the Textbook in Section 13-3.

TIMING

Both Demonstrations 29 and 30 should be done the day before students do Experiment 29. Total time for both demonstrations is about 40 minutes.

MATERIALS NEEDED

Quantities will depend upon the size of the class and the room. If an overhead projector is available, small quantities may be used and the demonstration projected. Amounts are for one demonstration.

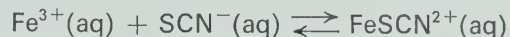
KSCN, 25 ml 0.00200 *M* (0.19 g KSCN/liter)
Fe(NO₃)₃, 0.5 ml 0.200 *M* [80 g Fe(NO₃)₃·9 H₂O/liter]
sample of KNO₃ solution
KSCN crystals
NaSCN crystals

Na₂HPO₄ crystals
1 beaker, 100 ml
4 test tubes, 25 × 200 mm
1 stirring rod
FeCl₃, 25 ml 0.1 *M* (27 g FeCl₃·6 H₂O/liter)

CLASSROOM PRESENTATION

Activity	Questions and Discussion
1. Put 25 ml 0.00200 <i>M</i> KSCN and 25 ml of water into a small beaker. Have 0.200 <i>M</i> Fe(NO ₃) ₃ in a dropper bottle. Give students the names of the reagents.	What ions are present (before mixing) ? $K^+(aq)$, $SCN^-(aq)$, $Fe^{3+}(aq)$, $NO_3^-(aq)$
2. Put three or four drops of Fe(NO ₃) ₃ into the beaker, and stir.	What possible explanation is there for the color change ? Color changes usually indicate a chemical change, and in this situation the new combinations are K^+ reacting with NO_3^- and Fe^{3+} with SCN^-
3. Have the solution of KNO ₃ ready.	Is there evidence that the color is not due to KNO ₃ ? Students may have used K^+ and NO_3^- in Experiment 17, but show a sample of KNO ₃ solution.

Conclusion: The color is most likely some combination of $\text{Fe}^{3+}(\text{aq})$ and $\text{SCN}^{-}(\text{aq})$. Investigation has shown the reaction to be



(Point out the conservation of charge.) Note that $\text{FeSCN}^{2+}(\text{aq})$ is the hydrated thiocyanoirron(III) ion.

4. Divide the FeSCN^{2+} solution in the beaker into four test tubes. Use the first as reference.

5. Indicate you intend to add KSCN.

6. Add KSCN crystals.

What will this do to the concentration of SCN^{-} ?

The $[\text{SCN}^{-}]$ should increase. (Here is a good opportunity to remind the student that brackets mean "concentration," expressed in moles/liter.)

What happened?

Color deepened.

What can we conclude from this intensification of color?

More FeSCN^{2+} must have formed.

How can this have happened, since no more Fe^{3+} was added?

There was some unreacted Fe^{3+} present. (In fact, the concentrations of SCN^{-} and of Fe^{3+} were about equal, with that of Fe^{3+} a bit in excess. But do not calculate these concentrations or tell the student the "correct" answer. Just accept the suggested explanation.)

7. Indicate you will add some $\text{Fe}(\text{NO}_3)_3$ solution to the third tube.

What will this do to the concentration of Fe^{3+} ?

$[\text{Fe}^{3+}]$ should increase.

8. Add 3 drops of 0.200 M $\text{Fe}(\text{NO}_3)_3$.

What happened?

Color deepened.

What can we conclude from this intensification of color?

More FeSCN^{2+} formed.

How can this have happened, since no more SCN^{-} was added?

There must have been some unreacted SCN^{-} present. You will probably find that you must supply this answer, since the student will be thinking that Fe^{3+} is in excess and that all the SCN^{-} reacted. Now point out that approximately equal amounts of the two ions were added. Ask how the color due to FeSCN^{2+} could have increased unless some SCN^{-} was available. Make a hypothesis. Some of all the species—products and reactants—are present at equilibrium.

9. After the above points are clearly made the following extension can be put to good use.

To the fourth tube add a few small crystals of Na_2HPO_4 , one at a time, with shaking between additions.

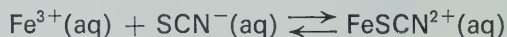
What could explain the reduction in color?

A reaction with FeSCN^{2+} , with Fe^{3+} , or SCN^- .

What ions were added, and, considering their charges, with which of the above might they react?

The Na^+ could react with SCN^- . The HPO_4^{2-} could react with Fe^{3+} or with FeSCN^{2+} .

The student does not have the information available but may be shown that NaSCN forms a colorless solution. He may then conclude that this would not affect the color. The general equation may be written and the effects of changing various concentrations discussed.



He may also be shown that HPO_4^{2-} forms a colorless solution with Fe^{3+} . Add a few crystals of Na_2HPO_4 to a FeCl_3 solution. The student might now be led to believe the color was removed by the formation of some colorless combination of Fe^{3+} and HPO_4^{2-} ions. The colorless species is FeHPO_4^+ .

Finally, outline the purpose of Experiment 29: To obtain concentration data for various equilibrium situations and to search for a regularity among them.

DEMONSTRATION 30

DEPTH AND COLOR

PURPOSE

To establish a relationship between height of a column of colored solution, and concentration, using a substance known to the student: tea.

TIMING

Should be done immediately after Demonstration 29 and the day before Experiment 29. About 15 minutes are needed.

MATERIALS NEEDED

- 1 tea bag
- overhead projector. See Lab Hint 1.
- 5 beakers, 100 ml

LAB HINT

1. The demonstration is written for use on an overhead projector. It is much more effective this way. Should one not be available, have students do the operations in pairs.

CLASSROOM PRESENTATION

- a) Make some drinkable tea, medium strength.
- b) Put equal amounts, about 50 ml, in each of two 100-ml beakers. On the projector screen these should have the same color.
- c) Use one as a control. To the other test beaker add water, commenting, "Watch the color get darker as the depth increases." It does not. Why? Perhaps color depends on total number of molecules the light passes through.
- d) To test this hypothesis, divide the diluted contents of the test beaker equally between it and another beaker. Same concentration, same height, same color. Mark the liquid level in one beaker. Remove 20 ml from it and replace with 20 ml of tea from a reserve. The height remains the same, the concentration increases, the color increases. Color depends directly on concentration.
- e) As a further check on this regularity, you may wish to reduce concentration. Split the contents of the unchanged control beaker from step d. Same concentration, same height, same color. Mark the liquid level in one beaker. Remove about $\frac{1}{3}$ of the liquid and replace it with an equal volume of water. The height remains constant, the concentration decreases, and the color decreases. Color depends directly on concentration.
- f) To test the effect of height on color, simply show deepening of color as height of a solution increases. Concentration remains the same, height increases (decreases), color increases (decreases). Color depends directly on height.
- g) This relationship can be expressed mathematically

$$\text{color} = k C_1 h_1$$

If the color is the same in two tubes containing the same chemicals, but different heights and concentrations, then

$$\text{color} = k C_1 h_1 = k C_2 h_2$$

or

$$C_1 h_1 = C_2 h_2$$

or

$$C_2 = \frac{C_1 h_1}{h_2}$$

- h) To check this formula intuitively, thereby giving the student a feeling it is correct, try the following. Consider two tubes in which the colors are identical.

If the height in one tube is twice that in the other ($2 h_1 = h_2$), then what can you say about C_1 and C_2 ? Since the color is the same intensity, isn't it reasonable that the concentration of one solution is twice the other? The stronger solution requires less height

($2 C_2 = C_1$). Then we can substitute in $\frac{C_1}{C_2} = \frac{h_2}{h_1}$ to get $\frac{2 C_2}{C_2} = \frac{2 h_1}{h_1}$: an identity.

Experiment 29

Chemical Equilibrium

PURPOSE AND SUMMARY

To determine an equilibrium constant experimentally, both as an introduction to the idea of equilibrium constants and to illustrate an important aspect of "searching for regularities."

The student prepares five solutions each containing a different amount of Fe^{3+} , but the same amount of SCN^- . He adjusts liquid depth in tube 1 to produce a color match with each of the four other tubes. This allows him to calculate the equilibrium concentration of the three ions in each of the four tubes. He tries equilibrium concentrations in three mathematical combinations to see if one will yield a regularity.

A computer program is provided to make all the calculations for this experiment. This optional feature can be used in several ways which are discussed on p. 166.

TIMING

This experiment must be completed before Section 13-3 is started. One full lab period is needed. Demonstrations 29 and 30 should precede the experiment.

MATERIALS NEEDED

per student

KSCN, 25 ml of 0.00200 *M* (1.94 g/100 ml, use 10 ml of this per liter of 0.00200 *M* solution). See Lab Hint 1.

$\text{Fe}(\text{NO}_3)_3$, acidified, 15 ml 0.200 *M* (80.8 g $\text{Fe}(\text{NO}_3)_3 \cdot 9 \text{H}_2\text{O}$ plus 40.0 ml conc. HNO_3 per liter). See Lab Hint 2.

distilled water in squeeze bottles

6 vials. See Lab Hint 3.

graduated cylinder, 25 ml
beaker, 100 ml
medicine dropper
ruler, metric

per class

light sources. See Lab Hint 4.

LAB HINTS

1. Dispensers, Nalgene "Pipettor," 5 ml, \$4.50 each, are excellent. Use them both as containers and dispensers. Plastic, disposable syringes, 5 ml, are a second best choice. They are available from hospital suppliers and cost about 10¢ each. They last several years if stored with plunger out of barrel. If neither of these is available, then graduated cylinders or pipets may be substituted.
2. Squeeze bottles are convenient for dispensing the $\text{Fe}(\text{NO}_3)_3$ solutions. You will probably save time by describing the dilution procedure for $\text{Fe}(\text{NO}_3)_3$ to ensure that students understand it. Have a student make a large chart of Figure 23 on butcher paper. Plastic, disposable syringes, 5 ml, are excellent aids for the dilutions.
3. Flat-bottomed vials (often used in biology) are needed for the color comparison. Fisher Scientific Co. specimen vials #3-330 (12 ml) are excellent. They eliminate errors due to

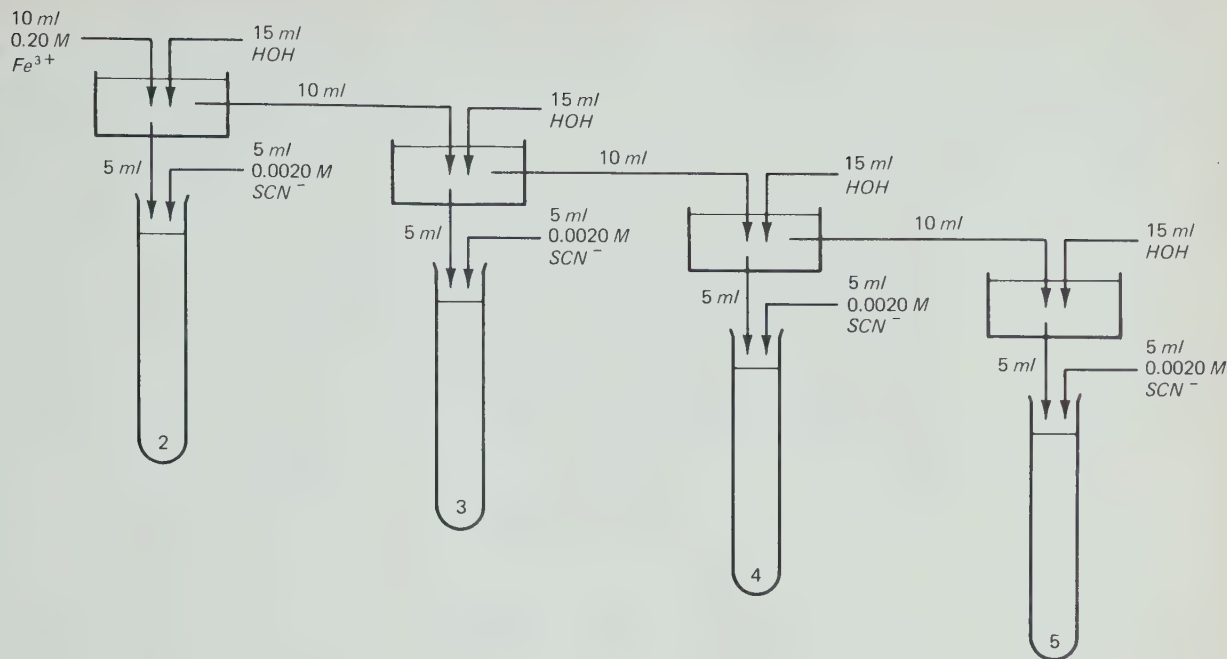


Figure 23 Sequence of operations to obtain solutions for tubes 2–5.

curvature and unevenness. If vials are not available, use the smallest diameter test tube you have that will hold 10 ml. A 13×100 mm tube or litmus vial will just barely hold this volume, hence mixing is messy.

Some prefer tall thin vials. The heights of liquid are great, reducing percent error in this measurement. Others prefer short fat vials. The larger area of color makes color comparison easier. Either will give good class averages. A useful rack (block) for keeping the tubes in order is shown in Figure 24. To make them, obtain blocks of soft wood $4\frac{3}{4}'' \times 2\frac{5}{8}'' \times 1\frac{1}{2}''$. Drill holes of suitable diameter with a spade drill. Let the point go through the bottom to provide drainage. Bevel and paint the tops. Number the holes 1 through 5. The extra hole is for a reserve tube for liquid removed from tube 1.

4. Matching the tubes for color intensity is difficult. The white paper background needs to be well lighted. If available, diffused light sources help some. A light box with a ground glass top may often be borrowed from either the commercial or the art department. You can make a light box by wiring a 150-watt bulb in a wooden box topped by a piece of window pane made translucent by rubbing it with 100-mesh grinding compound obtained from your auto shop or garage. Uniform, moderately bright light, as from a window, is satisfactory.

Two aids are:

- a) When colors seem close, remove a dropperful from the darker vial, return it drop by drop until the colors match.
 - b) Hold the tubes close together, and blink your eyes between "looks" to help avoid eye fatigue.
5. It is very important to suggest that students use exponential notation in recording concentrations and in doing calculations in this experiment. Many will need help in getting started on the calculations, especially in those involving the determination of concentration.

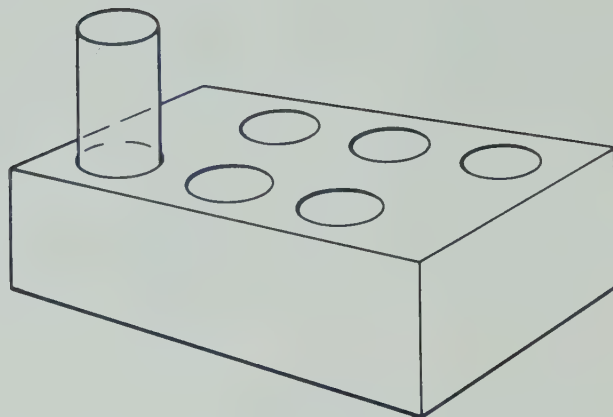


Figure 24 Block for holding vials.

PRELAB DISCUSSION

Demonstration 29 must precede this experiment; Demonstration 30 helps. Discuss Lab Hints 4 and 5.

If dry vials are not available for step a), have the student rinse the tube with some of the solution to be used in it.

Before the day of the experiment, assign the calculation of the initial concentrations of Fe^{3+} and SCN^- produced by dilution. During the lab period, check the student's calculation of $[\text{Fe}^{3+}]_{\text{initial}}$. If some are wrong, simply tell him so. There is not time during lab period to correct this. However, to do all the calculations with $[\text{Fe}^{3+}]_{\text{initial}}$ wrong is a bit disheartening.

Either a weekend or two school nights are needed to do the write-up.

Collect a carbon copy of each student's data before he leaves class.

SPECIAL FORMS

This experiment involves an unusual number of calculations. The student is not likely to organize these well enough. In addition, marking the experiment can be very time consuming if the papers are disorderly. To aid you and the student we suggest you prepare forms for this experiment. Also on pages 162–166 are some tables to speed your checking the students' calculations.

Name _____

Period _____ Date _____

Experiment 29**Chemical Equilibrium**

Before proceeding, you should be sure the assumption stated in the Lab Manual procedure is clearly understood. Writing it out helps.

	$\text{Fe}^{3+}(\text{aq}) + \text{SCN}^{-}(\text{aq}) \rightleftharpoons \text{FeSCN}^{2+}(\text{aq})$			NOTE: These moles are spread over 10 ml.
moles available before reaction	0.00100	0.000010	0	
moles present after equilibrium is reached	0.00099	0	0.000010	

DATA AND CALCULATIONS

Keep two significant figures throughout.
Depth of liquid in tube 1 needed to match color in other tubes.

Matching tube number	Height of liquid (mm)	
	Matching tube	Tube 1
2		
3		
4		
5		

Answer Question 9 here, after completing following pages.

Fill in the following table, using directions that follow it.

page 2

	A	B	C	D	E	F
Tube No.	Initial		Height Ratio	Equilibrium		
	$[\text{Fe}^{3+}]$	$[\text{SCN}^-]$	$\frac{\text{tube 1}}{\text{other tube}}$	$[\text{FeSCN}^{2+}]$	$[\text{Fe}^{3+}]$	$[\text{SCN}^-]$
1						
2						
3						
4						
5						

Column

A

Carefully follow through the process of diluting the original $0.200\text{ M Fe(NO}_3)_3$ solution. Clearly show the calculations needed for each determination. (Calculation 1 in Lab Manual.)

Tube 1

Tube 2

Tube 3

Tube 4

Tube 5

B

Each tube has 5 ml of 0.00200 M KSCN diluted by an equal volume of $\text{Fe(NO}_3)_3$ solution. All tubes then have the same initial concentration of SCN^- . What is it?

Column

page 3

C

Show height ratios here, $\frac{\text{mm tube 1}}{\text{mm other tube}}$, and quotients in the table.

D

To determine $[\text{FeSCN}^{2+}]_{\text{equilibrium}}$ in each tube, multiply the decimal ratio of heights by the equilibrium concentration of the thiocyanate ion in tube 1. (Calculation 3 in the Lab Manual.)

Tube 2 () () =

Tube 3 () () =

Tube 4 () () =

Tube 5 () () =

E

The $[\text{Fe}^{3+}]_{\text{equilibrium}}$ is the concentration left after that needed to form FeSCN^{2+} is subtracted. (Calculation 4 in the Lab Manual.)

Tube 2 — =

Tube 3 — =

Tube 4 — =

Tube 5 — =

F

The $[\text{SCN}^-]_{\text{equilibrium}}$ is the concentration of thiocyanate ion left after FeSCN^{2+} forms. (Calculation 5 in the Lab Manual.)

Tube 2 — =

Tube 3 — =

Tube 4 — =

Tube 5 — =

This information is now to be treated in several ways to find a mathematical relation that is constant for this set of equilibrium concentrations.

Give all answers to two significant figures.

page 4

Tube
No.**A****B****C**

	$\frac{[\text{FeSCN}^{2+}]}{[\text{Fe}^{3+}] + [\text{SCN}^-]}$	$\frac{[\text{Fe}^{3+}][\text{FeSCN}^{2+}]}{[\text{SCN}^-]}$	$\frac{[\text{FeSCN}^{2+}]}{[\text{Fe}^{3+}][\text{SCN}^-]}$
2	$\frac{(\quad)(\quad)}{(\quad) + (\quad)}$ =	$\frac{(\quad)(\quad)}{(\quad)}$ =	$\frac{(\quad)(\quad)}{(\quad)(\quad)}$ =
3	$\frac{(\quad)(\quad)}{(\quad) + (\quad)}$ =	$\frac{(\quad)(\quad)}{(\quad)}$ =	$\frac{(\quad)(\quad)}{(\quad)(\quad)}$ =
4	$\frac{(\quad)(\quad)}{(\quad) + (\quad)}$ =	$\frac{(\quad)(\quad)}{(\quad)}$ =	$\frac{(\quad)(\quad)}{(\quad)(\quad)}$ =
5	$\frac{(\quad)(\quad)}{(\quad) + (\quad)}$ =	$\frac{(\quad)(\quad)}{(\quad)}$ =	$\frac{(\quad)(\quad)}{(\quad)(\quad)}$ =
	largest _____ = smallest _____	_____ =	_____ =

Answer Question 8 here.

PRECAUTIONS

Observe the usual lab precautions.

POSTLAB DISCUSSION

This is best done by discussing the student write-ups.

SAMPLE DATA

The following data are given as a guide. You should use a set typical of your group. This can easily be obtained by arranging ratios of liquid heights so as to obtain medians. Such a display is also instructive to the student. There will be strong central tendencies that are reproducible year after year.

See Figure 25.

Depth of liquid in tube 1 needed to match color in other tubes.

Matching tube number	Height of liquid (mm)	
	Matching tube	Tube 1
2	45	41
3	45	36
4	46	31
5	44	19

RESULTS OF PROCESSING THE DATA

- 1) Carefully follow through the process of diluting the original $0.200\text{ }M\text{ Fe(NO}_3)_3$ solution to determine the initial concentration in moles of Fe^{3+} per liter, $[\text{Fe}^{3+}]_{\text{initial}}$, in each tube. This is the concentration before any Fe^{3+} has had a chance to react. Carry two significant figures through all calculations.

Column A on student form :

- Tube 1 $0.200\text{ }M$ but volume doubled, so $0.100\text{ }M$
 Tube 2 $0.20\text{ }M \times 10/25 = 0.080\text{ }M$, volume doubled, so $0.040\text{ }M$
 Tube 3 $0.080\text{ }M \times 10/25 = 0.032\text{ }M$, volume doubled, so $0.016\text{ }M$
 Tube 4 $0.032\text{ }M \times 10/25 = 0.0128\text{ }M$, volume doubled, so $0.0064\text{ }M$
 Tube 5 $0.0128\text{ }M \times 10/25 = 0.00512\text{ }M$, volume doubled, so $0.0026\text{ }M$

You might prefer the more rigorous treatment shown for tube 2.

A $0.20\text{ }M$ solution contains 0.00020 moles/ml.

10 ml were used, so 0.0020 mole is available.

This is spread throughout 25 ml so the solution is $\frac{0.0020\text{ mole}}{0.025\text{ liter}} = 0.080\text{ }M$.

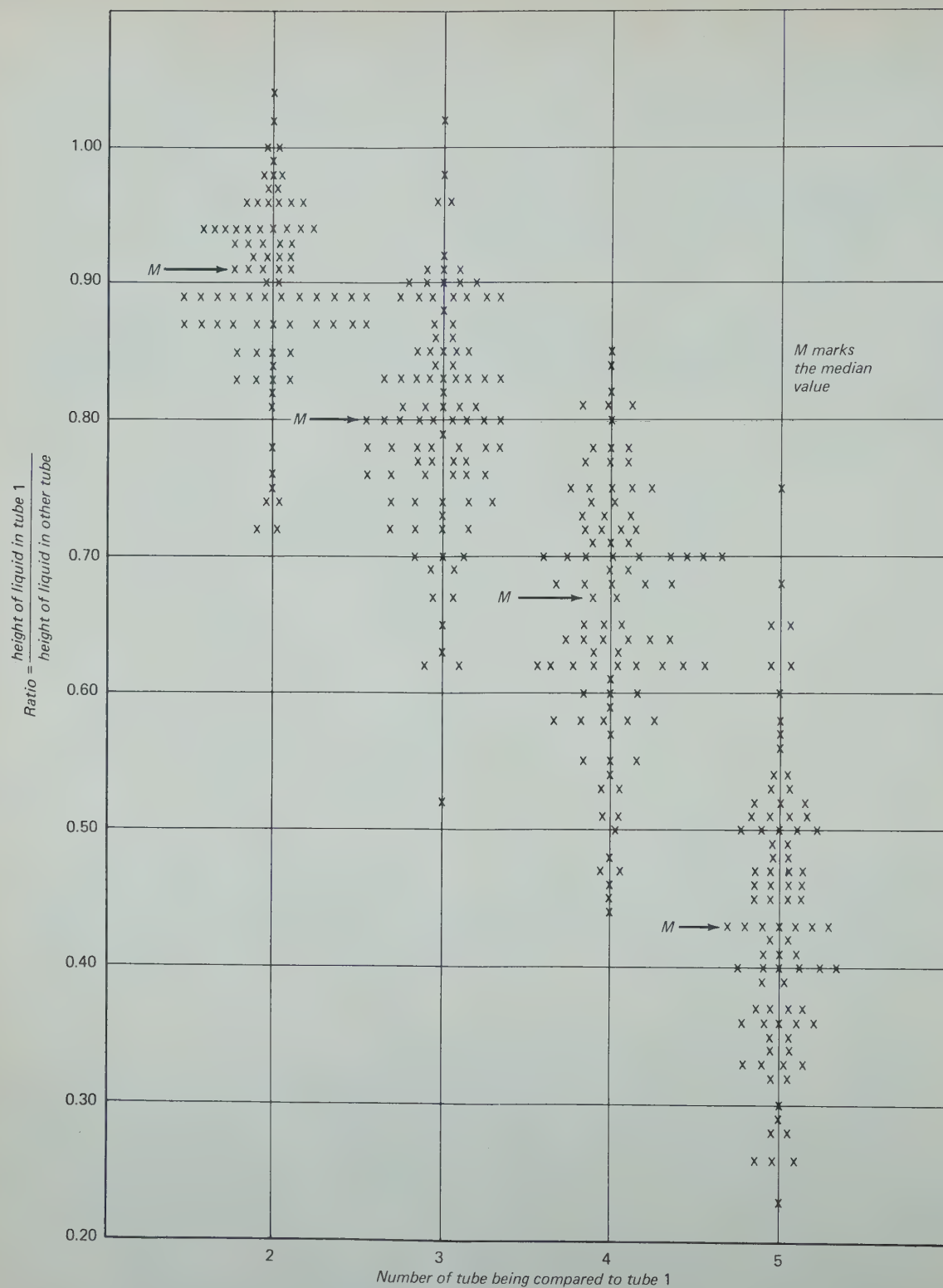


Figure 25 Distribution of ratio $\frac{\text{height of liquid in tube}}{\text{height of liquid in tubes 2-5}}$

This solution has its volume doubled by combination with an equal volume of SCN^- solution.

$$\text{Hence } [\text{Fe}^{3+}]_{\text{initial}} = \frac{0.080}{2} = 0.040 \text{ M.}$$

- 2) The solution in each tube was made by diluting 5.0 ml of 0.00200 M KSCN with 5.0 ml of a $\text{Fe}(\text{NO}_3)_3$ solution. Calculate $[\text{SCN}^-]_{\text{initial}}$.

Column B on student form :

$$[\text{SCN}^-]_{\text{initial}} = \frac{0.00200}{2} = 0.00100 \text{ M}$$

- 3) To calculate the equilibrium concentration of the colored species $[\text{FeSCN}^{2+}]_{\text{equilibrium}}$, recall that in tube 1 we assume all SCN^- was used to form FeSCN^{2+} . To determine $[\text{FeSCN}^{2+}]_{\text{equilibrium}}$ in the other tubes, use the relation $C_n = C_1 \frac{h_1}{h_n}$ where n refers to tubes 2, 3, 4, 5.

Column D on student form :

$$\left(\frac{[\text{FeSCN}^{2+}]_{\text{equilibrium}}}{\text{in tube 1}} \right) \left(\frac{h_1}{h_n} \right) = \frac{[\text{FeSCN}^{2+}]_{\text{equilibrium}}}{\text{in tube } n}$$

$$\text{Tube 2} \quad (1.0 \times 10^{-3})(0.91) = 9.1 \times 10^{-4} \text{ M}$$

$$\text{Tube 3} \quad (1.0 \times 10^{-3})(0.80) = 8.0 \times 10^{-4} \text{ M}$$

$$\text{Tube 4} \quad (1.0 \times 10^{-3})(0.67) = 6.7 \times 10^{-4} \text{ M}$$

$$\text{Tube 5} \quad (1.0 \times 10^{-3})(0.43) = 4.3 \times 10^{-4} \text{ M}$$

- 4) Determine $[\text{Fe}^{3+}]_{\text{equilibrium}}$ for each tube. This is $[\text{Fe}^{3+}]_{\text{initial}}$ reduced by the concentration needed to produce $[\text{FeSCN}^{2+}]_{\text{equilibrium}}$.

Column E on student form :

$$\text{Tube 2} \quad 0.040 - 0.00091 = 0.039 \text{ M}$$

$$\text{Tube 3} \quad 0.016 - 0.00080 = 0.015 \text{ M}$$

$$\text{Tube 4} \quad 0.0064 - 0.00067 = 0.0057 \text{ M}$$

$$\text{Tube 5} \quad 0.0026 - 0.00043 = 0.0022 \text{ M}$$

- 5) For each tube determine $[\text{SCN}^-]_{\text{equilibrium}}$. This is $[\text{SCN}^-]_{\text{initial}}$ reduced by the amount needed to produce $[\text{FeSCN}^{2+}]_{\text{equilibrium}}$.

Column F on student form :

$$\text{Tube 2} \quad 0.00100 - 0.00091 = 0.00009 \text{ M}$$

$$\text{Tube 3} \quad 0.00100 - 0.00080 = 0.00020 \text{ M}$$

$$\text{Tube 4} \quad 0.00100 - 0.00067 = 0.00033 \text{ M}$$

$$\text{Tube 5} \quad 0.00100 - 0.00043 = 0.00057 \text{ M}$$

Summary:

	A	B	C	D	E	F
Tube No.	Initial		Height Ratio $\frac{\text{Tube 1}}{\text{other tube}}$	Equilibrium		
	$[\text{Fe}^{3+}]$	$[\text{SCN}^-]$		$[\text{FeSCN}^{2+}]$	$[\text{Fe}^{3+}]$	$[\text{SCN}^-]$
1	0.100	0.00100		0.0010	0.099	0
2	0.040	0.00100	0.91	9.1×10^{-4}	3.9×10^{-2}	9.0×10^{-5}
3	0.016	0.00100	0.80	8.0×10^{-4}	1.5×10^{-2}	2.0×10^{-4}
4	0.0064	0.00100	0.67	6.0×10^{-4}	5.7×10^{-3}	3.3×10^{-4}
5	0.0026	0.00100	0.43	4.3×10^{-4}	2.2×10^{-3}	5.7×10^{-4}

- 6) The problem now is to see if there is a regularity among these equilibrium concentrations. Since regularities are often given a mathematical form, we will seek one here. Although there are many ways these equilibrium concentrations can be mathematically manipulated, we will try only three.

For each tube, calculate the values of the following three ratios, using the equilibrium concentration of each ion.

^c a) $\frac{[\text{FeSCN}^{2+}]}{[\text{Fe}^{3+}] + [\text{SCN}^-]}$
^a b) $\frac{[\text{Fe}^{3+}][\text{FeSCN}^{2+}]}{[\text{SCN}^-]}$
^c c) $\frac{[\text{FeSCN}^{2+}]}{[\text{Fe}^{3+}][\text{SCN}^-]}$

Use two significant figures in all calculations.

Tube No.	a	b	c
2	$\frac{[\text{FeSCN}^{2+}]}{[\text{Fe}^{3+}] + [\text{SCN}^-]}$ $\frac{(9.1 \times 10^{-4})}{(3.9 \times 10^{-2}) + (9.0 \times 10^{-5})}$ $= 2.3 \times 10^{-2}$	$\frac{[\text{Fe}^{3+}][\text{FeSCN}^{2+}]}{[\text{SCN}^-]}$ $\frac{(3.9 \times 10^{-2})(9.1 \times 10^{-4})}{(9.0 \times 10^{-5})}$ $= 3.9 \times 10^{-1}$	$\frac{[\text{FeSCN}^{2+}]}{[\text{Fe}^{3+}][\text{SCN}^-]}$ $\frac{(9.1 \times 10^{-4})}{(3.9 \times 10^{-2})(9.0 \times 10^{-5})}$ $= 2.6 \times 10^2$
3	$\frac{(8.0 \times 10^{-4})}{(1.5 \times 10^{-2}) + (2.0 \times 10^{-4})}$ $= 5.3 \times 10^{-2}$	$\frac{(1.5 \times 10^{-2})(8.0 \times 10^{-4})}{(2.0 \times 10^{-4})}$ $= 6.0 \times 10^{-2}$	$\frac{(8.0 \times 10^{-4})}{(1.5 \times 10^{-2})(2.0 \times 10^{-4})}$ $= 2.7 \times 10^2$
4	$\frac{(6.7 \times 10^{-4})}{(5.7 \times 10^{-3}) + (3.3 \times 10^{-4})}$ $= 1.1 \times 10^{-1}$	$\frac{(5.7 \times 10^{-3})(6.7 \times 10^{-4})}{(3.3 \times 10^{-4})}$ $= 1.2 \times 10^{-2}$	$\frac{(6.7 \times 10^{-4})}{(5.7 \times 10^{-3})(3.3 \times 10^{-4})}$ $= 3.6 \times 10^2$
5	$\frac{(4.3 \times 10^{-4})}{(2.2 \times 10^{-3}) + (5.7 \times 10^{-4})}$ $= 1.6 \times 10^{-1}$	$\frac{(2.2 \times 10^{-3})(4.3 \times 10^{-4})}{(5.7 \times 10^{-4})}$ $= 1.7 \times 10^{-3}$	$\frac{(4.3 \times 10^{-4})}{(2.2 \times 10^{-3})(5.7 \times 10^{-4})}$ $= 3.4 \times 10^2$

- 7) For each of the combinations a, b, and c in Question 6, calculate the value of the ratio :

$$\frac{\text{largest numerical value for tubes 2-5}}{\text{smallest numerical value for tubes 2-5}}$$

$\frac{\text{largest}}{\text{smallest}}$	$\frac{1.6 \times 10^{-1}}{2.3 \times 10^{-2}} = 7$	$\frac{3.9 \times 10^{-1}}{1.7 \times 10^{-3}} = 2.3 \times 10^2$	$\frac{3.6 \times 10^2}{2.6 \times 10^2} = 1.4$
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- 8) For the combination giving the smallest value for the ratio in Question 7, calculate the value of the ratio :

$$\frac{\text{largest concentration}}{\text{smallest concentration}}$$
 for each of the ions used

For combination c

Ion	$\frac{\text{largest concentration}}{\text{smallest concentration}}$
$[\text{Fe}^{3+}]$	$\frac{3.9 \times 10^{-2}}{2.2 \times 10^{-3}} = 1.8 \times 10$
$[\text{SCN}^-]$	$\frac{5.7 \times 10^{-4}}{9.0 \times 10^{-5}} = 6.3$
$[\text{FeSCN}^{2+}]$	$\frac{9.1 \times 10^{-4}}{4.3 \times 10^{-4}} = 2.1$

- 9) Restate in words the meaning of the mathematical combination (a, b, or c) which gave the smallest value of the ratio in Question 7.

The equilibrium concentration of the substances formed, divided by the product of the equilibrium concentrations of the reactants, gives a constant. Or, since the student will be reading the text at this time, he may say the *product* of the concentrations of the substances formed, even though in this situation only one substance is formed.

FORMS FOR USE IN MARKING REPORTS

To use Table 29-1, have a lab assistant check the carbon copies of the data, and write in the correct value for the ratio obtained from the body of the table. For example, if a student found 25 mm were needed in tube 1 to produce a color match with 47 mm in tube 3, his ratio, $\frac{\text{height of liquid in tube 1}}{\text{height of liquid in tube 3}}$, should be 0.53.

The table covers the values normally found with vials 26 mm $\times$ 63 mm [Fisher #3-330 (12 ml)]. If other tubes are used, ask the students to keep the heights in tubes 2-5 within the range of the table.

When the student write-ups are turned in, the lab assistant can quickly check the students' calculated ratios against the carbon copies.

Tables 29-2 through 5 contain results obtained for the three "regularities" tested. The tables contain the results the students will obtain with a given ratio of liquid heights—provided they make no errors. There is a separate table for each tube. Rather than check calculations for each tube each year, it is simpler to check only one. Use a different tube each year. Three significant figures are shown; students should report two.

Example: To check the calculated ratios for the data above, look in Table 29-3 (ratio of tube 1 to tube 3). The value 0.53 is not listed explicitly, but the value may be interpolated as halfway between those for 0.52 and 0.54. The students' calculated ratios should be 0.0333, 0.0174, and 73.0.

Table 29-1 Body of table gives ratio (in hundredths) of liquid heights $\frac{\text{tube 1}}{\text{tube 2 through 5}}$ needed to give equal color intensities.

[illegible]

Table 29-2 Results of manipulating equilibrium concentrations found in Tube 2.

HEIGHT RATIO			
$\frac{\text{mm tube 1}}{\text{mm tube 2}}$	$\frac{[\text{FeSCN}^{2+}]}{[\text{Fe}^{3+}] + [\text{SCN}^{-}]}$	$\frac{[\text{Fe}^{3+}][\text{FeSCN}^{2+}]}{[\text{SCN}^{-}]}$	$\frac{[\text{FeSCN}^{2+}]}{[\text{Fe}^{3+}][\text{SCN}^{-}]}$
0.30	0.00742	0.0170	10.8
2	793	187	11.8
4	843	204	13.0
6	894	223	14.2
8	944	243	15.5
0.40	0.00995	0.0264	16.8
2	105	287	18.3
4	110	311	19.9
6	115	337	21.5
8	120	365	23.4
0.50	0.0125	0.0395	25.3
2	130	428	27.4
4	135	463	29.7
6	140	502	32.3
8	146	544	35.0
0.60	0.0151	0.0591	38.1
2	156	643	41.4
4	161	700	45.2
6	166	764	49.3
8	172	836	54.0
0.70	0.0177	0.0917	59.4
2	182	0.101	65.5
4	187	0.112	72.5
6	192	0.124	80.7
8	198	0.139	90.4
0.80	0.0203	0.157	102
2	208	0.178	116
4	214	0.206	134
6	219	0.240	157
8	224	0.287	187
0.90	0.0230	0.352	230
2	235	0.449	294
4	240	0.612	401
6	246	0.937	615
8	251	1.91	1256
1.00	0.0256	∞	∞

Table 29-3 Results of manipulating equilibrium concentrations found in Tube 3.

HEIGHT RATIO			
mm tube 1 mm tube 3	$\frac{[\text{FeSCN}^{2+}]}{[\text{Fe}^{3+}] + [\text{SCN}^{-}]}$	$\frac{[\text{Fe}^{3+}][\text{FeSCN}^{2+}]}{[\text{SCN}^{-}]}$	$\frac{[\text{FeSCN}^{2+}]}{[\text{Fe}^{3+}][\text{SCN}^{-}]}$
0.30	0.0183	0.00673	27.3
2	196	738	30.0
4	208	807	32.9
6	221	880	36.0
8	234	957	39.2
0.40	0.0247	0.0104	42.7
2	260	113	46.5
4	273	122	50.5
6	286	132	54.8
8	299	143	59.5
0.50	0.0312	0.0155	64.5
2	326	168	70.0
4	339	181	75.9
6	353	196	82.4
8	366	213	89.6
0.60	0.0380	0.0231	97.4
2	393	251	106
4	407	273	116
6	421	298	126
8	435	326	139
0.70	0.0449	0.0357	152
2	463	393	168
4	477	434	186
6	491	483	208
8	505	540	233
0.80	0.0519	0.0608	263
2	534	0.0692	300
4	548	0.0796	346
6	563	0.0930	406
8	577	0.111	485
0.90	0.0592	0.136	596
2	607	0.173	762
4	622	0.236	1040
6	637	0.361	1600

Table 29-4 Results of manipulating equilibrium concentrations found in Tube 4.

HEIGHT RATIO			
mm tube 1 mm tube 4	$\frac{[\text{FeSCN}^{2+}]}{[\text{Fe}^{3+}] + [\text{SCN}^{-}]}$	$\frac{[\text{Fe}^{3+}][\text{FeSCN}^{2+}]}{[\text{SCN}^{-}]}$	$\frac{[\text{FeSCN}^{2+}]}{[\text{Fe}^{3+}][\text{SCN}^{-}]}$
0.30	0.0441	0.00261	70.3
2	473	286	77.4
4	506	312	85.0
6	539	340	93.1
8	572	369	102
0.40	0.0606	0.00400	111
2	640	433	121
4	675	468	132
6	710	506	143
8	745	546	156
0.50	0.0782	0.00590	170
2	818	637	184
4	845	688	200
6	892	743	218
8	929	804	237
0.60	0.0968	0.00870	259
2	0.101	943	282
4	0.105	0.0102	309
6	0.109	111	338
8	0.113	122	372
0.70	0.117	0.0133	409
2	0.121	146	453
4	0.125	161	503
6	0.129	179	562
8	0.134	199	631
0.80	0.138	0.0224	714
2	0.142	254	816
4	0.147	292	944
6	0.151	340	1109
8	0.156	405	1329
0.90	0.161	0.0495	1636

Table 29-5 Results of manipulating equilibrium concentrations found in Tube 5.

HEIGHT RATIO			
mm tube 1 mm tube 5	$\frac{[\text{FeSCN}^{2+}]}{[\text{Fe}^{3+}] + [\text{SCN}^-]}$	$\frac{[\text{Fe}^{3+}][\text{FeSCN}^{2+}]}{[\text{SCN}^-]}$	$\frac{[\text{FeSCN}^{2+}]}{[\text{Fe}^{3+}][\text{SCN}^-]}$
0.30	0.100	0.00096	186
2	0.108	107	206
4	0.116	116	228
6	0.125	126	251
8	0.134	136	276
0.40	0.143	0.00146	303
2	0.152	158	332
4	0.162	170	364
6	0.172	182	398
8	0.182	196	435
0.50	0.192	0.00210	476
2	0.203	225	521
4	0.214	242	570
6	0.226	260	624
8	0.238	279	684
0.60	0.250	0.00300	750
2	0.263	323	824
4	0.276	348	907
6	0.289	376	1000
8	0.304	408	1110
0.70	0.318	0.00443	1230
2	0.333	483	1370
4	0.349	529	1530
6	0.365	583	1720
8	0.382	645	1950
0.80	0.400	0.00720	2220

COMPUTER PROGRAM FOR TREATING DATA

Students often hear of the marvels of modern computers. This experiment provides a chance for them to use their own data, or have it used, in a computer. The following program offers several teaching possibilities.

1. You may have a student interested in trying to write a program. You can suggest he use data from this experiment and the following program as a check. *Note* that it is not unique and that someone else will surely use different symbols.
2. The program can be shown to a class along with a discussion of its usefulness.

3. A computer may be available in the school, local college, or industrial laboratory. If so, a student (or parent) might calculate the results for the entire class using this program. Such a use should not replace the student's own calculation but be contrasted with it as an indication of the time saving that is possible. A sample printout is given after the program.

This program is written in FORTRAN IV, a language especially developed for scientific problems. The name, derived from *formula translation*, indicates a strong feature. FORTRAN statements look very much like algebraic equations.

It is not expected that you would be able to learn programming from this short example. However, it will be useful if you have some understanding of the special terms, whose meanings are explained below.

DIMENSION tells the computer how much storage space is needed for each variable. For example, `RATIO(5)` means five values of the variable named `RATIO` will be needed. The student measures four values and the computer later supplies the unit value that would have been found if the solution in tube 1 had been compared with itself. This allows more convenient calculation.

COMMON alerts the computer to items that will be needed in subroutines. The computer will keep track of where these are stored.

READ directs the computer to take information from cards or tape.

FORMAT specifies the arrangement of data either coming into the computer or how it is to be arranged as it is printed out.

IF denotes one mechanism by which the computer can take alternative paths. This first example (just after the second READ statement) is used to tell the computer whether it has completed all the sets of data. As long as there are additional data sets, the computer will find a nonzero value of FEB, the starting concentration of Fe^{3+} . When all the data have been treated, the "flag" card with zero in this position will cause this IF statement to direct the computer to statement 55 and stop the program. There is another example three lines after statement 8. Concentrations are to be lowered by a factor of 2 or 2.5. This second IF statement selects the proper one.

FORMAT statements can be quite complex, as those at the end of the program show. You will have to consult a text to find an explanation of these. On the other hand you need to know what one of them means in order to put the data into the computer. `FORMAT (2F10.6,8F5.1)` can be translated as follows.

2 is just a multiplier—there are two of whatever is specified up to the comma.

F indicates the number occurring in this field has a decimal point;

10 specifies that ten spaces are left on the card;

6 means the decimal point has 6 spaces to its right.

Thus the "field" would look like this xxx.xxxxxx and could accommodate a number such as 102.774582. The field need not be filled but it must not be overfilled. The two F10.6 fields are used for starting concentrations. In the same way 8F5.1 means there are eight fields of the form xxx.x. These are for the eight heights measured by the student.

DO is the beginning of a DO LOOP. Its action is best described by an example. Note this set of statements beginning 3 lines after statement 5.

```

      DO 8 I = 1,4
      J = I + 1
      8  RATIO(J) = HS(I)/HT(I)

```

The first line declares that the computer is to do all the statements from this point on to and including that specified as 8. $I = 1,4$ shows that these statements will be done four times, first with $I = 1$, then 2 and so on. $J = I + 1$ tells the computer to compute the value of J as one greater than I . The third line sets up the calculation of the J th value of RATIO as the ratio of $HS(I)$ divided by $HT(I)$. HS and HT are matching heights of standard and test solutions. To be specific, the computer first sets $I = 1$, finds $J = 2$ and $RATIO(2) = HS(1)/HT(1)$. Then, having reached the end of the DO loop at statement 8, the computer returns; sets $I = 2$, finds $J = 3$ and $RATIO(3) = HS(2)/HT(2)$. This cycle is repeated for $I = 3$ and $I = 4$. At this point the computer recognizes it has done the loop the required number of times and skips to the next statement after the ending one; i.e., the one after 8 in this case.

CALL signifies that the computer is to make use of a subroutine specified by the next word, QUOT in this case. Subroutines are used as a convenient way to repeat the same calculation

WRITE is the command to give out the computed data according to the format specified. Often the write statements become complicated in order to arrange the printout in the desired manner.

To learn more about FORTRAN programming, consult a beginner's text such as:

Fortran II and IV for Engineers and Scientists. H. Golde, Macmillan Co., New York, 1966, \$4.50.
A Guide to Fortran IV Programming. D. D. McCracken, John Wiley and Sons, Inc., New York, 1965, \$3.95.

EQ.CON - EFN SOURCE STATEMENT - IFN(S) -

```

C      THIS PROGRAM COMPUTES EQUILIBRIUM CONSTANT EXPRESSIONS FOR
C      EXPERIMENT 29 IN CHEMISTRY-EXPERIMENTS AND PRINCIPLES.
C      IT REQUIRES STARTING CONCENTRATIONS OF FE3+ AND SCN- PLUS THE
C      8  VALUES OF HEIGHTS OF LIQUIDS WHEN COLORS MATCH.
C      DEFINITIONS
C      A=(FESCN)/(FE)+(SCN)
C      B=(FE)(FESCN)/(SCN)
C      C=(FESCN)/(FE)(SCN)
C      FEB,SCNB =STARTING CONCENTRATIONS BEFORE DILUTION
C      FEA,SCNA=CONCENTRATIONS AFTER DILUTION
C      FESCN,FEE,SCNE=EQUILIBRIUM CONCENTRATIONS
C      HS,HT=HEIGHTS IN TUBES CONTAINING STANDARD AND TEST SOLUTIONS
C      WHEN THEY MATCH
C      QUOT=NAME OF SUBROUTINE TO FIND MAX AND MIN VALUES AND
C      COMPUTE THEIR RATIO
C      RA,RB,RC  =MAX/MIN FOR EXPRESSIONS A,B,C
C      RFE,RSCN,RFS = MAX/MIN VALUES OF EQUILIBRIUM CONC. OF
C      FE3+,SCN-, AND FESCN2+
C      RATIO=HS/HT
C
C      DATA INPUT CARDS-TWO PER STUDENT
C      TITLE-ANY 80 CHARACTERS-NO FORMAT

```

```

C      DATA=FEB,SCNB,HS(4 VALUES), HT(4VALUES) IN 2F10.6,8F5.1
C      END OF DATA IS SIGNALLED BY BLANK CARD FOLLOWED BY A CARD WITH
C      0.0 FOR FEB
C
C      NOTE ALL REMARKS DENOTED BY C AT LEFT ARE IGNORED BY COMPUTER
C      THEY ARE COMMENTS FOR THE HUMAN READER.
C
C      PROGRAM STARTS
C
C      DIMENSION RATIO(5),A(5),B(5),C(5),FEA(5),SCNA(5),FEE(5),SCNE(5),
C      IFESCN(5),TITLE(16),HS(4),HT(4)
C      COMMON A,B,C
C  COMPUTER READS A TITLE CARD
C      3 READ(5,4)(TITLE(K),K=1,16)
C      4 FORMAT(16A5)
C  COMPUTER READS A DATA CARD
C      READ(5,5)FEB,SCNB,HS,HT
C      IF(FEB.EQ.0.0) GO TO 55
C  THE PREVIOUS STATEMENT FINDS THE BLANK CARD AND STOPS OPERATION
C      5 FORMAT(2F10.6,8F5.1)
C  CALCULATE RATIOS OF HEIGHTS AND SET THE FIRST RATIO EQUAL TO 1.0
C      RATIO(1)=1.00
C      DO 8 I=1,4
C          J=I+1
C          8 RATIO(J)=HS(I)/HT(I)
C  CALCULATE INITIAL AND EQUILIBRIUM CONCENTRATIONS
C      DO 10 I=1,5
C          IF(I.GT.1) GO TO 6
C          FFA(I)=FEB/2.0
C          GO TO 7
C      6 J=I-1
C          FEA(I)=FEA(J)/2.5
C      7 SCNA(I)=SCNB/2.0
C          FESCN(I)=SCNA(I)*RATIO(I)
C          SCNE(I)=SCNA(I)-FESCN(I)
C
C      10 FEE(I)=FEA(I)-FESCN(I)
C  CALCULATE VARIOUS POSSIBLE CONSTANTS A,B, AND C
C      DO 20 I=1,5
C      20 A(I)=FESCN(I)/(FEE(I)+SCNE(I))
C          DO 21 I=2,5
C              B(I)=(FEE(I)*FESCN(I))/SCNE(I)
C      21 C(I)=FESCN(I)/(FEE(I)*SCNE(I))
C  CHOOSING LARGEST AND SMALLEST VALUES AND GETTING THEIR RATIO
C      CALL QUOT (A,RA,5,2)
C      CALL QUOT (B,RB,5,2)
C      CALL QUOT (C,RC,5,2)
C      CALL QUOT (FEE,RFE,5,2)
C      CALL QUOT (SCNE,RSCN,5,2)
C      CALL QUOT (FESCN,RES,5,2)
C
C  PRINTING RESULTS - THE FIRST WRITE STATEMENT PRINTS THE TITLE
C  THE SECOND WRITES THE HEADINGS AND FILLS IN THE TABLE
C  THE THIRD WRITE STATEMENT ADDS THE FINAL LINE WITH THE RATIO

```



```

C  OF LARGEST TO SMALLEST VALUE
  WRITE(6,38)(TITLE(K),K=1,16)
38  FORMAT(1H1,16A5)
  WRITE(6,39)
39  FORMAT(1H0,2X,7HTUBE NO,3X,7HFE INT.,3X,8HSCN INT.,3X,
15HRATIO,5X,5HFESCN,4X,6HFE EQ.,4X,8HSCN. EQ., 5X,1HA,11X,1HB,11X,
21HC)
  WRITE(6,40)(I,FEA(I),SCNA(I),RATIO(I),FESCN(I),FEE(I),
1 SCNE(I),A(I),B(I),C(I),      I=1,5)
40  FORMAT((1H0,5X,I1,5X,E9.2,1X,E9.2,3X,F5.2,3X,2(E9.2,1X),
14(E9.2,3X))/)
  WRITE(6,45)RFS,RFE,RSCN,RA,RB,RC
45  FORMAT(1H0,41X,4HRFS=,F5.1,1X,4HRFE=,F5.1,1X,5HRSCN=,F5.1,3X,3HRA=
1,F5.1,4X,3HRB=,F5.1,4X,3HRC=,F5.1,4X)
50  CONTINUE
  GO TO 3
C  THE PREVIOUS STATEMENT SENDS THE COMPUTER BACK FOR THE NEXT SET OF DATA
55  CONTINUE
  RETURN
  END

```

```

SUBROUTINE QUOT(X,RX,N,J)
  DIMENSION X(N)
  XMIN=X(J)
  XMAX=X(J)
  DO 25 I=J,N
    XMIN=AMIN1(X(I),XMIN)
    XMAX=AMAX1(X(I),XMAX)
25  RX=XMAX/XMIN
  RETURN
  END

```

0.16E - 03 means 0.16 X 10 ⁻³
--

PRINTOUT FOR ONE STUDENT

WILLIAM BREITHAUPT

TUBE NO	FE INT.	SCN INT.	RATIO	FESCN	FE EQ.	SCN. EQ.
1	0.10E 00	0.10E-02	1.00	0.10E-02	0.99E-01	0.00E-38
2	0.40E-01	0.10E-02	0.84	0.84E-03	0.39E-01	0.16E-03
3	0.16E-01	0.10E-02	0.70	0.70E-03	0.15E-01	0.30E-03
4	0.64E-02	0.10E-02	0.50	0.50E-03	0.59E-02	0.50E-03
5	0.26E-02	0.10E-02	0.33	0.33E-03	0.22E-02	0.67E-03

A	B	C
0.10E-01	-0.00E-19	-0.00E-20
0.21E-01	0.21E 00	0.14E 03
0.45E-01	0.36E-01	0.16E 03
0.78E-01	0.59E-02	0.17E 03
0.11E 00	0.11E-02	0.22E 03
RA= 5.2	RB=191.8	RC= 1.6

Experiment 30

Applying Le Chatelier's Principle

PURPOSE AND SUMMARY

Le Chatelier's Principle is illustrated, using the CrO_4^{2-} — $\text{Cr}_2\text{O}_7^{2-}$ equilibrium. The student investigates the effect of H^+ and OH^- on CrO_4^{2-} and $\text{Cr}_2\text{O}_7^{2-}$ and of H^+ on OH^- and *vice versa* (Part I). He also investigates the effect of H^+ and OH^- on $\text{BaCrO}_4(\text{s})$ (Part II). In optional Part III, the effect of CH_3COOH , HNO_3 , $\text{Ca}(\text{OH})_2$, H_2SO_4 , KOH , $\text{C}_2\text{H}_5\text{OH}$, and $\text{NH}_3(\text{aq})$ on the $\text{CrO}_4^{2-} \longrightarrow \text{Cr}_2\text{O}_7^{2-}$ equilibrium are studied separately.

TIMING

Immediately after Section 13-7. Parts I and II can both be done in one lab period. Part III (Optional) requires another.

MATERIALS NEEDED

per student

Roman numbers indicate the parts in which chemicals are needed.

About 2 ml each of the following solutions (these should be in dropper bottles) :

K_2CrO_4 , 0.1 M (19.4 g K_2CrO_4 /liter) all
 $\text{K}_2\text{Cr}_2\text{O}_7$, 0.1 M (29.4 g $\text{K}_2\text{Cr}_2\text{O}_7$ /liter) all
 plastic thumb protectors, all

NaOH, 1 *M* (40 g NaOH/liter) I, II
HCl, 1 *M* (85.5 ml conc. HCl/liter) I, II
Ba(NO₃)₂, 0.1 *M* (26.1 g Ba(NO₃)₂/liter) II
CH₃COOH, 1 *M* (56.9 ml glacial acid/liter) III
HNO₃, 1 *M* (64 ml conc. HNO₃/liter) III
H₂SO₄, 1 *M* (55.5 ml conc. H₂SO₄/liter) III
KOH, 1 *M* (56.1 g KOH/liter) III
NH₃(aq), 1 *M* (66.3 ml conc. reagent/liter) III
Ca(OH)₂, saturated (approx. 3.6 g Ca(OH)₂/liter) III
C₂H₅OH III

6 test tubes, 13 × 100 mm. See Lab Hint 2.

LAB HINTS

1. Have many sets of reagents available in dropper bottles in order that time will not be wasted by students waiting for reagents.
2. Yellow BaCrO₄ stains are easily removed from tubes by dipping them in dilute CH₃COOH.

PRELAB DISCUSSION

Little discussion of this experiment is needed. Remind the students to consider the reacting species involved in each step of the experiment. To mix chemicals in a test tube, cover it with a plastic square and hold this in place with a finger. The student should be aware of the questions he will have to answer under Processing the Data.

PRECAUTIONS

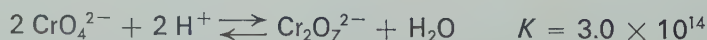
Observe the usual laboratory precautions. Wear safety glasses and apron.

POSTLAB DISCUSSION

Question 5 for Part II should provide the principal basis for discussion. Place emphasis on the H⁺ + OH⁻ equilibrium. This is a good opportunity to mention the importance of the equilibrium between H⁺ and OH⁻ in biological systems as well as in many industrial processes.

SAMPLE DATA

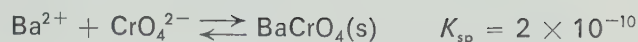
PART I



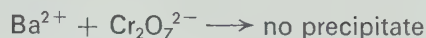
- a) CrO₄²⁻, yellow; Cr₂O₇²⁻, orange.
- b) When NaOH solution is added, Cr₂O₇²⁻ becomes yellow. The color then matches that of the CrO₄²⁻.
- c) When HCl solution is added, CrO₄²⁻ becomes orange. The color then matches that of the Cr₂O₇²⁻.
- d) Adding NaOH solution to (c) reverses the color change.
- e) Adding HCl solution to (b) reverses the color change.

PART II

f) A yellow, dense precipitate forms ;



g) No precipitate forms ;



h) The precipitate dissolves, and the yellow solution changes to orange.

i) The color changes to yellow, and a precipitate forms.

j) In step (c) add sodium hydroxide to reverse the change, and in step (d) add hydrochloric acid.

k) A heavy precipitate forms with CrO_4^{2-} , and a light precipitate forms with $\text{Cr}_2\text{O}_7^{2-}$.

PART III (OPTIONAL)

The color changes show that CrO_4^{2-} is converted to $\text{Cr}_2\text{O}_7^{2-}$ with CH_3COOH , HNO_3 , and H_2SO_4 . The reverse happened with $\text{Ca}(\text{OH})_2$, KOH , and NH_3 . No change is observed with $\text{C}_2\text{H}_5\text{OH}$.

RESULTS OF PROCESSING THE DATA

QUESTIONS FOR PART I

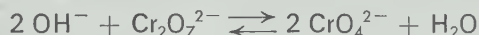
1. What can you conclude about the change $2 \text{CrO}_4^{2-} \longrightarrow \text{Cr}_2\text{O}_7^{2-}$ and its dependence upon hydrogen ions, H^+ , as noted in steps (c) and (e)? Produce a balanced equation by adding the proper number of H^+ ions and H_2O molecules to the appropriate sides of the equation.

The addition of H^+ to the reaction causes the equilibrium concentration of $\text{Cr}_2\text{O}_7^{2-}$ to increase :



2. What can you conclude about the reverse change $\text{Cr}_2\text{O}_7^{2-} \longrightarrow 2 \text{CrO}_4^{2-}$ and its dependence on hydroxide ions, as noted in steps (b) and (d)? Produce a balanced equation by adding the proper number of OH^- ions and H_2O molecules to the appropriate sides of the equation.

The addition of OH^- causes the equilibrium concentration of CrO_4^{2-} to increase :



QUESTIONS FOR PART II

3. From your observation in step (k), what can you conclude about the relative equilibrium concentrations of CrO_4^{2-} ion in each of the solutions 0.1 M $\text{K}_2\text{Cr}_2\text{O}_7$ and 0.1 M K_2CrO_4 ?

There are CrO_4^{2-} ions in each solution, but the concentration in $\text{K}_2\text{Cr}_2\text{O}_7$ is much less than in K_2CrO_4 .

4. Use the equations you balanced in Questions 1 and 2 of Part I to explain the results you obtained in steps (h), (i), and (j).

In (f) and (g) we learn that $\text{BaCr}_2\text{O}_7(\text{s})$ is much more soluble than is BaCrO_4 . Since adding OH^- favors the formation of CrO_4^{2-} , we find that BaCrO_4 forms when OH^- is present. Adding H^+ favors the formation of $\text{Cr}_2\text{O}_7^{2-}$, and we find that adding H^+ dissolves this precipitate.

5. Make a statement summarizing your results with the chromate ion-dichromate ion equilibrium, which includes the application of the Principle of Le Chatelier.

Using the equations



we can see that adding H^+ to the first is the same as removing OH^- and that adding OH^- to the second is the same as removing H^+ . Thus the equilibrium is shifted to counteract the imposed change—to form more CrO_4^{2-} if either OH^- is added or H^+ is removed and to form more $\text{Cr}_2\text{O}_7^{2-}$ when the reverse happens.

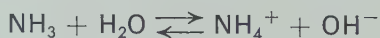
QUESTIONS FOR PART III

6. a) Which substances in solution caused the color to change from that of the $\text{Cr}_2\text{O}_7^{2-}$ ion to that of the CrO_4^{2-} ion?
b) Which substances in solution caused the reverse color change?
a) $\text{Ca}(\text{OH})_2$, KOH , NH_3 .
b) CH_3COOH , HNO_3 , H_2SO_4 .

7. What ionic species do the solutions you listed in Question 6(a) have in common? Answer the same question for the solutions listed in Question 6(b).

All the solutions in 6(a) contain OH^- , and those in 6(b) contain H^+ .

8. Give an explanation of the results you noted
a) when ethyl alcohol, $\text{C}_2\text{H}_5\text{OH}$, was added.
b) when the solution of aqueous ammonia, NH_3 , was added.
a) Since no change was noted with $\text{C}_2\text{H}_5\text{OH}$ we may conclude that it produces no H^+ or OH^- in solution.
b) The observed change was that given by compounds containing OH^- , thus we may conclude that this change is evidence for the reaction



9. On the basis of your conclusion made in Question 7, predict some additional substances which in solution might have the same effect on the CrO_4^{2-} — $\text{Cr}_2\text{O}_7^{2-}$ equilibrium as those you listed in Questions 6(a) and 6(b).

Solutions having the same effect as those in 6(a) would be any which have OH^- in solution. Among the possibilities are NaOH , LiOH , RbOH , and CsOH . The student may also mention $\text{Ba}(\text{OH})_2$ and $\text{Ca}(\text{OH})_2$ but may recognize that these would give precipitates of $\text{BaCrO}_4(\text{s})$ and $\text{CaCrO}_4(\text{s})$.

Solutions having the same effect as those in 6(b) would be any which have H^+ in solution. Possibilities include oxalic acid, sulfurous acid, the halogen acids, and phosphoric acid.

DEMONSTRATION 31

SPONTANEOUS ENDOTHERMIC REACTION

PURPOSE

To show an equilibrium reaction that favors products even though it is extremely endothermic.

TIMING

This should come after a discussion of factors which determine equilibrium, Section 13-15 in the Textbook. The demonstration takes just a few minutes.

MATERIALS NEEDED

- 1 conical flask, 250 ml
- 1 stopper, solid rubber to fit flask
- 1 wooden block
- 31.5 grams $\text{Ba}(\text{OH})_2 \cdot 8 \text{H}_2\text{O}$ as fine crystals
- 15.2 grams NH_4SCN as fine crystals

CLASSROOM PRESENTATION

Mix crystalline $\text{Ba}(\text{OH})_2 \cdot 8 \text{H}_2\text{O}$ with crystalline NH_4SCN in a conical flask, stopper it, and swirl the mixture for a few seconds until a solution begins to form. Place the flask on a wet wooden block (surface of block wet with water). Let the students make observations without any discussion. See *Follow-up Section*.

DISCUSSION

The reaction is observed to be spontaneous and favors products at equilibrium even though it is endothermic. According to the relation

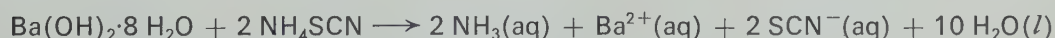
$$\Delta G = \Delta H - T\Delta S$$

if the change in free energy, ΔG , is negative the reaction will proceed spontaneously. In this reaction, entropy is increased as the solid substances combine to form a solution (more random state).

We can estimate a ΔH for the reaction by making several assumptions:

- 1) that $\text{Ba}(\text{SCN})_2$ is formed and is dissolved to form aquated ions; and
- 2) that NH_3 is mostly present in the stoppered flask as $\text{NH}_3(\text{aq})$.

Then we can write the equation



to represent the reaction. The ΔH of formation values from handbooks are:

Reactants

Ba(OH) ₂ ·8 H ₂ O(s)	=	-800 kcal/mole	-800 kcal
NH ₄ SCN(s)	=	-20 kcal/mole	-40 kcal
total for reactants			-840 kcal

Products

NH ₃ (aq)	=	-19 kcal/mole	-38 kcal
SCN ⁻ (aq)	=	+17 kcal/mole	+34 kcal
Ba ²⁺ (aq)	=	-129 kcal/mole	-129 kcal
H ₂ O(l)	=	-68 kcal/mole	-680 kcal
total for products			-813 kcal

$$\Delta H \text{ for reaction} = -813 \text{ kcal} - (-840 \text{ kcal}) = +27 \text{ kcal}$$

The reaction is endothermic since ΔH is positive and clearly spontaneous. Thus ΔG is negative and $T\Delta S$ must be larger than ΔH . The energy needed for the reaction to proceed is drawn from the environment and the water on the block of wood freezes.

FOLLOW-UP

Ask the students to account for the observed behavior of the system by considering the following questions:

1. Why did the water freeze?
2. Where did the energy go?
3. Which is favored at equilibrium, reactants or products?
4. What did you observe that would indicate the system has changed in free energy?
5. Which of the factors ΔH or $T\Delta S$ initially must have been greater?

REFERENCE

"A Spontaneous Endothermic Reaction," Submitted by Professor Hambly to *Journal of Chemical Education*.

Aqueous Acids and Bases



Experiment 31 shows that the main reacting species in neutralization reactions are H^+ and OH^- . Equilibrium considerations permit ranking acids according to their ability to donate protons. Titration and the use of indicators is shown by Experiments 32 and 33.

Schedule of Experimental Work

TEXT SECTION	EXPERIMENTAL WORK	FOR DETAILS SEE PAGE
14-1 to 14-3	Expt. 31	178
	Demo. 32	181
	Demo. 33	183
14-4	Expt. 32	184
14-5 to 14-8	Expt. 33	188
14-9 to 14-11		

Experiment 31

Heat of Acid-Base Reactions

PURPOSE AND SUMMARY

To measure the energy released when solutions of acids and bases are mixed, and to correlate energy released with the equation for the reaction involving only the predominant reacting species.

The students measure the temperature changes which occur as 50 ml of an acidic solution is mixed with 50 ml of a basic solution. Each student or pair uses two or more combinations. By sharing class data they obtain comparative data for all possible combinations of the four acidic with the three basic solutions.

TIMING

Fits best after Section 14-3 in the Textbook. Note that Demonstration 32, p. 184, of this guide is to be done after this experiment, but in the same period if possible.

The students will need about twenty minutes to obtain data from two different combinations. About 15 minutes are needed for the demonstration of electrical conductivity.

MATERIALS NEEDED

per student or pair

- 1 beaker, 100 ml
- 1 Styrofoam cup, 8 oz
- 1 thermometer, -20° to 150°C

for the class

Each trial requires 50 ml of an acid and 50 ml of a base.

acidic and basic solutions. See Lab Hint 1.

CH_3COOH , 2.0 *M* (113.8 ml conc. reagent/liter of solution)

$\text{NH}_3(\text{aq})$, 2.2 *M* (147 ml conc. reagent/liter of solution)

HCl , 2.0 *M* (171.0 ml conc. reagent/liter of solution)

HNO_3 , 2.0 *M* (128.0 ml conc. reagent/liter of solution)

KOH , 2.2 *M*, (123 grams solid/liter of solution)

NaOH , 2.2 *M* (88 grams solid/liter of solution)

H_2SO_4 , 1.0 *M* (56.0 ml conc. reagent/liter of solution)

graduated cylinders or volumetric flasks, 50 ml. See Lab Hint 2.

LAB HINTS

1. Since the results of each combination are to be compared one with another, it is essential to have the concentration of the solutions as accurate as possible. The acidic solutions should be made up from fresh bottles of concentrated reagent. For best results they should also

be titrated, after thorough mixing, with a standard base. Adjust the concentration as necessary to obtain the indicated concentrations. The basic solutions are made 2.2 *M* so they will have at least 0.1 mole of base per 50 ml of solution. Sodium hydroxide and potassium hydroxide pellets react with carbon dioxide in the air forming a carbonate, thus some of the solid weighed is carbonate rather than hydroxide. Making the basic solutions slightly more concentrated makes it possible to obtain very reproducible results using the acidic concentrations as the controlling factor.

All solutions should be prepared at least one day before they are to be used to allow them to come to room temperature. Be *sure* to mix the solutions thoroughly.

2. *Dispensing the solutions.* With a number of classes doing the experiment, the required volumes may be rather large. It is a good idea to use a siphon arrangement or pump dispensing arrangement in large stock bottles. If you place a labeled 50-ml volumetric flask or graduated cylinder by each stock bottle, you can facilitate the dispensing of the solutions. Tie a string to the flask or graduated cylinder to prevent its use with other solutions. Thus they will not have to be rinsed after each use.

PRELAB DISCUSSION

Point out that all reactions to be studied take place in solution. Tell the students to mix the solutions quickly and to measure the highest temperature reached immediately after mixing. They should use the same thermometer to measure all temperatures. Remind them to rinse and dry the thermometer before transferring it to another solution. Since the reaction is rapid, there will be little energy lost to the cup and to the atmosphere. Some students, however, may be interested in determining more precisely the change in temperature of the mixture. You may want to have them record time-temperature data every minute and then plot the data to obtain a graph like the one shown in Figure 26. Recording time-temperature data for several minutes before mixing and several minutes after permits one to extrapolate backward to the time of mixing to obtain a more precise Δt . *Note:* the exact time of mixing must be recorded.

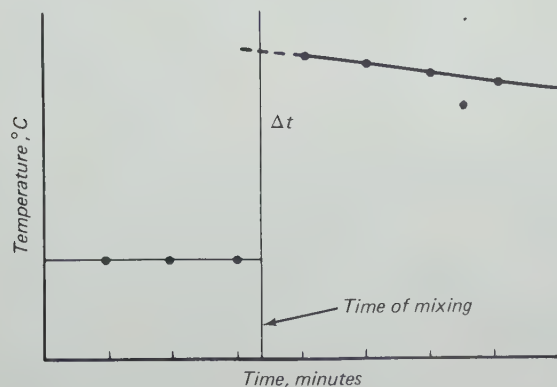


Figure 26
Time-temperature graph
showing extrapolation to obtain Δt
for the reaction.

PRECAUTIONS

In addition to the usual laboratory precautions, warn the students about keeping even dilute sulfuric acid off clothing. Have students wear safety glasses and aprons.

POSTLAB DISCUSSION

Obtain averages of data from an entire class (or from several classes). Have students compare ΔH values obtained in this experiment to those obtained in Experiment 26. The principal point of the experiment concerns the similarities rather than the differences in the molar heat of reaction. The student should realize that the reaction H^+ with OH^- is the important one and that other ions do not matter. Deviations from ΔH values obtained for strong acid-strong base reactions can be rationalized in terms of energy needed to break bonds in the predominant reacting species other than H^+ and OH^- . The higher values obtained using sulfuric acid are perhaps partially due to the heat of hydration of the sulfate ions.

SAMPLE DATA

Change in temperature, Δt , using 50 ml of each solution.

	$\Delta t, ^\circ\text{C}$		
	NaOH	KOH	$\text{NH}_3(\text{aq})$
HCl	13.8	14.5	12.6
H_2SO_4	15.6	15.9	14.7
HNO_3	13.9	14.3	14.1
CH_3COOH	13.0	13.4	12.4

RESULTS OF PROCESSING THE DATA

- 1) Write net ionic equations for each of the twelve reactions. Write the formulas for the strong electrolytes in ionic form and the formulas for the weak electrolytes in molecular form to indicate the reacting species present. Include ΔH for each reaction. Review the calculations you made in Experiment 26 to determine the molar heat produced by the reaction $\text{H}^+ + \text{OH}^- \rightleftharpoons \text{H}_2\text{O} + \text{heat energy}$. Make a similar calculation to obtain the heat energy involved *per mole* of H^+ used in each reaction you performed in this experiment. Neglect any energy lost to the environment.

Combination	Equation	ΔH , kcal/mole H^+ used
1	$\text{H}^+ + \text{OH}^- \rightleftharpoons \text{H}_2\text{O}$	13.8
2	$\text{H}^+ + \text{OH}^- \rightleftharpoons \text{H}_2\text{O}$	14.5
3	Since $\text{NH}_3(\text{aq})$ reacts with water $\text{NH}_3(\text{aq}) + \text{H}_2\text{O} \rightleftharpoons \text{NH}_4\text{OH} \rightleftharpoons \text{NH}_4^+ + \text{OH}^-$ there are two possibilities $\text{NH}_3(\text{aq}) + \text{H}^+ \rightleftharpoons \text{NH}_4^+$ or $\text{H}^+ + \text{OH}^- \rightleftharpoons \text{H}_2\text{O}$	12.6
4	$\text{H}^+ + \text{OH}^- \rightleftharpoons \text{H}_2\text{O}$	15.6
5	$\text{H}^+ + \text{OH}^- \rightleftharpoons \text{H}_2\text{O}$	15.9
6	$\text{NH}_3(\text{aq}) + \text{H}^+ \rightleftharpoons \text{NH}_4^+$ or $\text{H}^+ + \text{OH}^- \rightleftharpoons \text{H}_2\text{O}$	14.7
7	$\text{H}^+ + \text{OH}^- \rightleftharpoons \text{H}_2\text{O}$	13.9
8	$\text{H}^+ + \text{OH}^- \rightleftharpoons \text{H}_2\text{O}$	14.3
9	$\text{NH}_3(\text{aq}) + \text{H}^+ \rightleftharpoons \text{NH}_4^+$ or $\text{H}^+ + \text{OH}^- \rightleftharpoons \text{H}_2\text{O}$	14.1
10	$\text{CH}_3\text{COOH} + \text{OH}^- \rightleftharpoons \text{CH}_3\text{COO}^- + \text{H}_2\text{O}$	13.0
11	$\text{CH}_3\text{COOH} + \text{OH}^- \rightleftharpoons \text{CH}_3\text{COO}^- + \text{H}_2\text{O}$	13.4
12	$\text{NH}_3(\text{aq}) + \text{CH}_3\text{COOH} \rightleftharpoons \text{NH}_4^+ + \text{CH}_3\text{COO}^-$ or $\text{CH}_3\text{COOH} + \text{OH}^- \rightleftharpoons \text{CH}_3\text{COO}^- + \text{H}_2\text{O}$	12.4

- 2) Using class data, what regularities do you observe in the ΔH values? What do all the equations have in common? Base your generalization on averages of class data.

The ΔH is similar for all the reactions. The ΔH 's for reactions involving $\text{NH}_3(\text{aq})$ all tend to be a little lower than those involving the other bases. The ΔH 's for reactions involving CH_3COOH tend to be the lowest for the reactions of any one base.

- 3) Propose an explanation for the differences and similarities in the ΔH values based upon the energy needed to make and break chemical bonds.

Weak acids are predominantly molecular in aqueous solution; thus energy is required to break the O—H bonds. This could account for the lower values obtained with acetic acid-strong base reactions. The high values obtained for the sulfuric acid reactions could be partially due to the hydration energy of the sulfate ion.

DEMONSTRATION 32

ELECTRICAL CONDUCTIVITY OF ACIDS AND BASES

PURPOSE

To demonstrate relative conductivity, which is used as a basis for inferring degree of ionization. This, in turn, is used to define "weak" and "strong" electrolytes.

TIMING

This demonstration should be done after Experiment 31, but *before* the questions are assigned. The experiment and demonstration should be done in one period. About 15 minutes are needed.

MATERIALS NEEDED

conductivity apparatus described in Demonstration 21, p. 99.

50 ml each of the 7 solutions used in Experiment 31, diluted to 0.1 *M*

50 ml H_2SO_4 diluted to 0.02 *M*

50 ml $\text{Ba}(\text{OH})_2$, 0.02 *M* (0.4 g/100 ml)

8 small beakers or bottles to contain solutions

CLASSROOM PRESENTATION

PART I

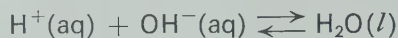
Test the conductivity of each of the solutions used in Experiment 31 (diluted to 0.1 *M*), and test the conductivity of distilled water. Have each student record the results in his notebook, classifying each solution as a good conductor (strong electrolyte) or as a poor conductor (weak electrolyte).

PART II

Test the conductivity of the following :

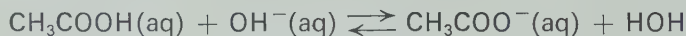
A. Strong acid with a strong base.

1. Recall the conductivity of 0.1 *M* solutions of HCl and NaOH just measured.
2. Mix equal volumes of the two solutions together, and test the conductivity of half of the resulting solution.
3. Have the students write the overall equation and then the net ionic equation for the reaction



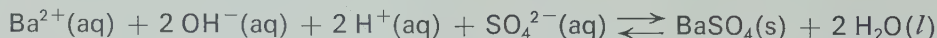
B. Weak acid with a strong base.

1. Recall the conductivity of 0.1 *M* solutions of acetic acid and NaOH just measured.
2. Mix equal volumes of the two solutions together, and test the conductivity of half of the resulting solution.
3. Write the net ionic equation



C. Acid with a base to form a slightly soluble salt.

1. Test the conductivity of a 0.02 *M* solution of sulfuric acid and of a 0.02 *M* solution of Ba(OH)₂.
2. Titrate the 0.02 *M* Ba(OH)₂ solution with the 0.02 *M* H₂SO₄, using phenolphthalein indicator. At the same time, observe the relative conductivity of the solution. When moles of Ba(OH)₂ equal moles of H₂SO₄ the light will go out. After that, slowly add an excess of acid, and follow the conductivity.
3. Write the net ionic equation



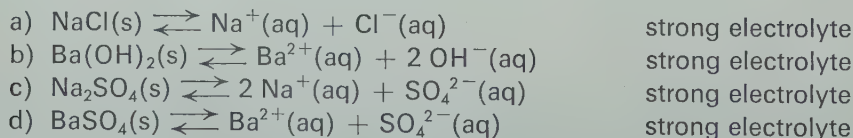
FOLLOW-UP

Discuss the equations and exercises after the students have had a chance to complete them.

EXERCISES

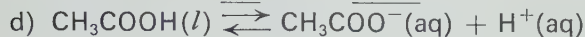
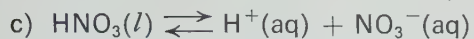
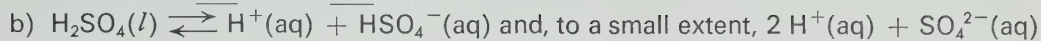
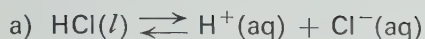
1. Write equations describing the equilibrium existing in saturated water solutions of the following compounds. Indicate which solutions are strong electrolytes.

- a) NaCl
- b) Ba(OH)₂
- c) Na₂SO₄
- d) BaSO₄



2. Write equations for the dissociation of the following acids in aqueous solution. Underline the substances favored in the equilibrium.

- a) $\text{HCl}(l)$
- b) $\text{H}_2\text{SO}_4(l)$
- c) $\text{HNO}_3(l)$
- d) $\text{CH}_3\text{COOH}(l)$



DEMONSTRATION 33

TITRATION

PURPOSE

To illustrate the technique of titration, showing where care is needed, and where it is not.

TIMING

About 45 minutes are needed; should precede Experiment 32.

MATERIALS NEEDED

2 burets
cleaning equipment
1 ring stand
1 double buret clamp
2 bottles, 250 ml
2 conical flasks, 250 ml

phenolphthalein solution. See Experiment 32.
distilled water in squeeze bottle
1 graduated cylinder, 50 ml
1 graduated cylinder, 10 ml
HCl, 6 *M*
NaOH, 6 *M*

PROCEDURE

Clean and assemble the burets. Prepare about 100 ml of 0.2 *M* solutions of HCl and NaOH. Dilute 3.3 ml of 6 *M* solution up to 100 ml. Emphasize these measurements need not be made with great care. It is important to develop a sense of what is important and what is not. Mix the solutions carefully. Rinse the cleaned burets with a bit of the solution that is to be used in them. One way to emphasize the effectiveness of several rinses each made with a small volume, compared to a single rinse made with a large volume, is to pose the following problem. Assume that one milliliter is left behind after each rinse. What volume, in a single rinse, is needed to be as effective as three rinses of 9 ml each? During the *Postlab Discussion* you can give the answer—999 ml, or 1 liter. Each 9 ml solution gives a 10/1 dilution; therefore three give a 1000/1 dilution.

Fill the burets and demonstrate the elimination of air from the tip. Keep the labelled acid bottle near the acid buret and the base bottle near the other for identification. Add about 50 ml of distilled water to a clean conical flask, and a few drops of indicator. Record buret readings (no need to have them on zero) and start to titrate. Show the reaching of an endpoint after only a few drops of each solution have been used. Explain why this is an unsatisfactory place to end the titration (large percent error). Continue to add solutions until about 20 ml of one solution is used.

At this point, use another flask, containing only water, to demonstrate the difference between swirling and shaking. The latter will usually result in some liquid leaving the container.

Return to the titration flask, rinse down the inside, and finish the titration.

Record reading, refill burets, empty and prepare a conical flask as before and perform another titration. Point out that this one can be done more rapidly than the first. The approximate ratio can now be seen. Rapidly add about 20 ml of the solution requiring the larger volume in the first titration, then titrate to an endpoint. Three titrations can easily be done in one period. Have the students calculate the (ml acid)/(ml base) ratio for each titration. It is not difficult to obtain three identical results—to three significant figures.

OPTIONAL EXPERIMENT

After seeing this demonstration, the students benefit from doing an acid-base titration in which they prepare their own solutions as suggested under *Procedure*. Have them determine the (ml acid)/(ml base) ratio for three trials. This is good preparation for Experiment 32.

Experiment 32

Acid-Base Titration

PURPOSE AND SUMMARY

To give the student some experience in acid-base titration, as well as additional experience in quantitative analysis.

A NaOH solution is first standardized against an HCl solution of known molarity, and is then used to titrate a known mass of an unknown acid. The grams of acid that will react with one mole of NaOH is calculated.

TIMING

The base can be standardized in $\frac{1}{2}$ period. The unknown solid acid can be titrated the next day. The experiment should be done before Section 14-5.

MATERIALS NEEDED

per student

2 burets

1 ring stand

1 double buret clamp

1 conical flask, 250 ml

1 bottle, small mouth, 250 ml. See Lab Hint 1.

per class

Have a sufficient number of the following available so that students do not spend too much time looking for them.

wash bottle with distilled water

hot detergent solution

buret brushes

phenolphthalein solution in dropper bottles (1 g/100 ml 50% ethanol)

NaOH solution. See Lab Hint 2.

HCl solution. See Lab Hint 3.

unknown solid acids. See Lab Hint 4.

LAB HINTS

1. Students can bring pint bottles from home. They should be clean, sealable, and handy to pour from.
2. The NaOH solution may be handled in various ways.

- a) Have each student prepare his own. Six titrations with 25 ml burets will require about 150 ml. To make 200 ml of 0.2 *M* NaOH, dilute 7 ml of 6 *M* NaOH.
- b) Prepare a large amount for the students. Thirty students will need 6 liters (1.6 gal). Dissolve 48 g NaOH(s) per 6 liters for 0.2 *M*. Equip a large container with siphon. If students take reserve to their benches, prepare a very large excess.
- c) The NaOH solution may be made in several different concentrations so that each student will not have the same. Make up 8 liters of 0.3 *M* NaOH (96 g/8 liters). Save 2 liters to be used directly, and dilute the rest as follows:

To 1800 ml 0.3 *M* NaOH, add 200 ml distilled water to give 0.27 *M*.

To 1600 ml 0.3 *M* NaOH, add 400 ml distilled water to give 0.24 *M*.

To 1400 ml 0.3 *M* NaOH, add 600 ml distilled water to give 0.21 *M*.

To 1200 ml 0.3 *M* NaOH, add 800 ml distilled water to give 0.18 *M*.

These dilutions yield five different 2 liter solutions of base from 0.18 to 0.3 *M*. Two liters will accommodate ten students. Most standard acids are purchased in 2 liter bottles, which are convenient for storing and dispensing these solutions. Make sure the solutions are thoroughly mixed.

3. The standard 0.200 *M* acid should be made up in a large batch to insure uniformity. Measure carefully 171 ml of concentrated (11.7 *M*) HCl (*fresh* bottle). Place it in a 1 liter volumetric flask, and fill to 1 liter mark with distilled H₂O. Empty this into a large container, and add 9 more liters of distilled water. Mix until stirring produces no schlieren. Acid diluted in this manner need not be standardized. Five-gallon water jugs are convenient for mixing and storing this large volume. Have students fill burets directly from a siphon-equipped storage bottle, or prepare a large excess. Thirty students using 25-ml burets will need 2500 ml.

The acid may be standardized against a sodium hydroxide solution that has been standardized against a solid acid.

4. Suitable solid acids for use as unknowns are:

Acid	Grams Acid Needed for 25 ml of 0.2 <i>M</i> NaOH
potassium hydrogen sulfate (KHSO ₄ , molar mass = 136.2 g)	0.7
potassium acid tartrate (KHC ₄ H ₄ O ₆ , molar mass = 188.2 g)	0.9
potassium acid phthalate (KHC ₈ H ₄ O ₄ , molar mass = 204.2 g)	1.0
potassium acid oxalate (KHC ₂ O ₄ , molar mass = 128.1 g)	0.6

The corresponding sodium compounds can also be used. The unknown solid acids should be prepared in advance by placing 2 to 3 grams of the acid in a small container. Thus with even the most dilute base, the student will probably not have to use more than 25 ml in the titration. Potassium hydrogen tartrate probably won't dissolve until it is converted during titration. Inform the students of this so that they don't waste time trying to dissolve it.

A different unknown for each student can easily be prepared as follows. Use a 50 ml, automatic-zero pipet (Fisher 13-685, 50 ml, about \$12) to add 50.00 ml distilled water to one small-mouth bottle for each student. Use an automatic-zero 25 or 50 ml buret (Fisher 3-750, about \$15) to add between 12 and 24 ml of 1.20 *M* HCl to each bottle. The molarity need not be accurately known. Make about 3 liters of it (30.8 ml conc. HCl/3 liters solution). Assume this is 1.20 *M*, and standardize a base against it. Use this base to standardize the acid the students will use when they standardize the base they make.

The molarity of each student's unknown acid can be read off a 20" × 24" graph. To make the graph, calculate *M* of the diluted acid for each ml of 1.20 *M* acid added between 12 and 24 ml. For example, if you put 12 ml of 1.20 *M* acid into a total volume of 62.00 ml, the concentration of this diluted unknown is (12.00)(1.20)/62.00 = 0.232 *M*. Locate these points. Have someone bend a meterstick to aid in drawing a curve through the points.

It is interesting to plot the student obtained values on this graph. Most will fall very near the curve.

PRELAB DISCUSSION

Point out the usefulness of titration as a technique for quantitative determinations. Demonstrate a titration. See Demonstration 33.

PRECAUTIONS

Remind students of the dangers of handling acids and bases. Have them wear safety glasses and aprons.

POSTLAB DISCUSSION

Collect and post results from Question 7 to show the precision with which such titration may be made.

SAMPLE DATA AND RESULTS OF PROCESSING THE DATA

PART I **Standardizing the base** $M_{\text{acid}} = 0.200$

	Trial 1		Trial 2		Trial 3	
	acid	base	acid	base	acid	base
final ml	23.61	21.68	24.19	23.75	21.52	22.52
initial ml	0.34	0.02	0.11	1.39	0.49	2.86
ml used	23.27	21.66	24.08	22.36	21.03	19.66

PART II **Titration unknown acid**

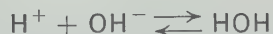
Trial	1	2	3
grams acid initial	12.63	12.15	11.53
grams acid final	12.15	11.53	10.83
grams acid used	0.48	0.62	0.70
ml base final	16.23	21.45	24.10
ml base initial	0.17	0.08	0.10
ml base used	16.06	21.37	24.00

- 1) From the concentration given and volume used, calculate the number of moles of hydrochloric acid involved in each titration of Part I.

$$\text{Moles} = (\text{moles/liter})(\text{liters}) \quad M_{\text{acid}} = 0.200$$

Trial number	1	2	3
ml acid used	23.27	24.08	21.03
moles acid used	$(0.200)(0.02327)$ $= 0.004654$	$(0.200)(0.02408)$ $= 0.004816$	$(0.200)(0.02103)$ $= 0.004206$

- 2) Use the equation for the reaction, to determine how many moles of base are used per mole of acid in Part I.



One mole of base reacts with one mole of acid.

- 3) Using the relation in Question 2, determine the moles of base used in each trial.

Same as the last numbers in each column, Question 1.

- 4) Calculate the molarity of the base. Compute the average of the molarities found for each of the three trials.

$$\text{moles base} = (\text{moles base/liter})(\text{liters}) = M_{\text{base}} (\text{liters})$$

Trial number	1	2	3
$M_{\text{base}} = \frac{\text{moles base}}{\text{liters}}$	$\frac{0.004654}{0.02166}$	$\frac{0.004816}{0.02236}$	$\frac{0.004206}{0.01966}$
	$= 0.215$	$= 0.216$	$= 0.214$
Average $M_{\text{base}} = \frac{0.215 + 0.216 + 0.214}{3} = 0.215$			

- 5) For each trial in Part II, calculate the mass of the solid unknown acid that will react with one mole of the base. Determine the average of these values.

Trial number	1	2	3
moles base = moles acid	$(0.215)(0.0161)$ $= 0.00346$	$(0.215)(0.0214)$ $= 0.00460$	$(0.215)(0.0240)$ $= 0.00516$
g acid/mole base	$\frac{0.48}{0.00346}$ $= 139$	$\frac{0.62}{0.00460}$ $= 135$	$\frac{0.70}{0.00516}$ $= 136$
Average g/mole base $= \frac{139 + 135 + 136}{3} = 137$			

- 6) Using the formula of the acid given by your teacher, calculate the theoretical value for the mass of the acid that will react with one mole of the base.

For potassium hydrogen sulfate, KHSO_4 , the molar mass is 136.2 g. This amount will react with one mole of base.

- 7) Determine the percent error, using the value calculated in Question 6 as the accepted value.

$$\left(\frac{137 - 136}{136} \right) (100) = 0.74\% \text{ error}$$

Experiment 33

Determination of $[\text{H}^+]$ Using Indicators

PURPOSE AND SUMMARY

This experiment has four aims: To observe the effect of acidic and basic solutions on selected indicators; to determine the $[\text{H}^+]$ of various solutions using student prepared standard indicator solutions for comparison; to determine the equilibrium constant for a weak acid; and to perform a simple titration.

Working in pairs for Parts I and II, one student prepares a set of acidic standards using Methyl Orange and Orange IV indicators, while the other prepares a set of basic standards using Indigo

Carmine and Alizarin Yellow R. These solutions are prepared from successive dilutions of 0.1 *M* HCl and 0.1 *M* NaOH solutions. See Lab Hint 1.

In Part III the students independently determine the $[H^+]$ of an unknown solution using both sets of standards for comparison. In Part IV they determine the $[H^+]$ of a dilute solution of a weak acid which is used to calculate the equilibrium constant for the acid. In Part V they do a simple titration using 0.1 *M* solutions of NaOH, HCl, and CH_3COOH .

TIMING

This experiment should be done after the students have studied Section 14-8 in the Textbook.

The experiment requires one full period if the reagents are conveniently located and the students understand what they are to do. The standards fade in color overnight, and although they may be brought back by adding another drop of indicator, it is much better to do the entire experiment in one day.

MATERIALS NEEDED

per pair of students

- 22 test tubes, 13 × 100 mm
- 2 graduated cylinders, 10 ml
- 2 medicine droppers or pipets. See Lab Hint 2.
- 2 test tube racks
- 1 vial of litmus paper
- 1 marking pencil or 16 labels
- Stock solutions. See Lab Hint 1.
- 15 ml HCl, 0.1 *M* (8.6 ml conc. reagent liter)
- 15 ml NaOH, 0.1 *M* (4.0 grams NaOH liter)
- 15 ml acetic acid, 0.1 *M* (5.7 ml conc. reagent liter)
- 15 ml acetic acid, 1.0 *M* (56.9 ml conc. reagent liter)

per class

- Indicators, 8 sets in dropper bottles. See Lab Hint 3.
- Orange IV (0.1 gram/100 ml water)
- Methyl Orange (0.1 gram/100 ml water)
- Indigo Carmine (0.25 gram/100 ml 50% ethanol)
- Alizarin Yellow R (0.1 gram/100 ml water)
- phenolphthalein (1.0 gram/100 ml 50% ethanol)
- Unknowns, 5 ml per student. See Lab Hint 4.
- acetic acid, about 0.1 *M*, for rinsing slippery fingers

LAB HINTS

1. Directions are given for each student to prepare dilutions for 0.01, 0.001, and 0.0001 *M* acidic and basic solutions. If you are short of time, you may wish to supply some or all of these. For example, you may wish to provide 0.1 and 0.001 *M* solutions of acid and base. The student would then prepare 0.01 and 0.0001 *M* solutions. This method also assures somewhat more uniform concentrations. To prepare 0.001 *M* solutions, dilute 10 ml of

0.1 *M* solutions up to 1 liter.

- If medicine droppers are used as pipets, the students should calibrate them for a volume of one milliliter. Have them fill a 10 ml graduate cylinder to the 10 ml mark. Then draw one ml of the solution into the medicine dropper and make a mark to indicate 1 milliliter. Ten ml plastic syringes are useful here and can be used in place of both the medicine dropper and the 10 ml graduated cylinder. They are available from hospital suppliers and are inexpensive.
- The indicators selected are those that give distinct color changes in the ranges we are particularly interested in. There are many other indicators possible, but if too many sets of standards are prepared the students become confused. The ranges for those suggested are:

	$[H^+]$ range	pH range	Color change
Orange IV (Tropaeolin OO)	500 to 6×10^{-4}	1.3–3.2	R–Y
Methyl Orange (Orange III, Tropaeolin D)	800 to 4×10^{-5}	3.1–4.4	R–OY
Alizarin Yellow R	100 to 1×10^{-12}	10–12	Y–R
Indigo Carmine (sodium indigo disulfonate)	25 to 1×10^{-13}	11.6–13	B–Y

(R = red ; Y = yellow ; OY = orange-yellow ; B = blue.)

The Indigo Carmine should be dark blue when freshly prepared. If it fades in use, add more. If these indicators are not available, others may be substituted (see handbooks).

- The H^+ concentration of the following unknowns is within the range of the indicators discussed in the foregoing :

Indicator	$[H^+]$	pH	Acid	To Make 100 ml Solution Use
Orange IV	3×10^{-2}	1.5	0.05 <i>M</i> oxalic acid	0.63 g $C_2H_2O_4 \cdot 2 H_2O$
	10^{-2}	2	0.03 <i>M</i> citric acid	0.7 g $C_6H_3O_7 \cdot H_2O$
	3×10^{-3}	2.5	1 <i>M</i> acetic acid	5.7 ml conc. CH_3COOH
	10^{-3}	3	0.1 <i>M</i> potassium hydrogen phthalate and 0.1 <i>M</i> hydrochloric acid	2 g $C_6H_4COOHCOOK$ and 10 ml 1 <i>M</i> HCl
Methyl Orange	3×10^{-4}	3.5	0.03 <i>M</i> potassium hydrogen tartrate	0.63 g $C_3H_5O_4COOK$
	10^{-4}	4	0.05 <i>M</i> potassium hydrogen phthalate	1 g $C_6H_4COOHCOOK$
	3×10^{-5}	4.5	0.1 <i>M</i> sodium acetate and acetic acid	1.36 g $CH_3COONa \cdot 3 H_2O$ and 10 ml 1 <i>M</i> CH_3COOH
	10^{-10}	10	0.1 <i>M</i> sodium hydrogen carbonate and 0.1 <i>M</i> sodium carbonate	0.84 g $NaHCO_3$ and 1.24 g $Na_2CO_3 \cdot H_2O$
Alizarin	3×10^{-11}	10.5	saturated magnesium hydroxide	about 0.001 g MgO
Yellow R	3×10^{-12}	11.5	0.05 <i>M</i> sodium carbonate	0.62 g $Na_2CO_3 \cdot H_2O$
Indigo	10^{-12}	12	0.03 <i>M</i> trisodium phosphate	1.3 g $Na_3PO_4 \cdot 12 H_2O$
Carmine	3×10^{-13}	12.5	saturated calcium hydroxide	about 0.1 g $Ca(OH)_2$

PRELAB DISCUSSION

Tell the students that there are many dyes whose colors change with $[H^+]$. We have selected some that are convenient for the concentrations used in this experiment. Emphasize the need

for careful measurement in preparing the standards. Mention also the need for careful rinsing of glassware before use.

If students' fingers become slippery because of contact with base, have them rinse their fingers in 0.1 *M* acetic acid solution. Ask the students why not use dilute HCl?

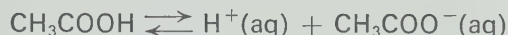
PRECAUTIONS

Observe the usual laboratory precautions. Have students wear safety glasses and aprons.

POSTLAB DISCUSSION

RESULTS OF PROCESSING THE DATA

- 1) Calculate the equilibrium constant for the equilibrium reaction involving an aqueous solution of the weak acid, acetic acid.



Use the value for the hydrogen ion concentration determined in Step 1. You may assume that the concentration of the acetate ion is equal to the $[\text{H}^+]$ and that the concentration of acetic acid is essentially the concentration of the solution you used, either 0.1 *M* or 1.0 *M*. The $[\text{H}^+]$ for the 0.1 *M* acetic acid corresponded to that in the 0.001 *M* HCl. Therefore the $[\text{H}^+]$ is 10^{-3} , and the $[\text{CH}_3\text{COO}^-]$ is also 10^{-3} . The $[\text{CH}_3\text{COOH}]$ is 10^{-1} . (Remind the students of the conductivity demonstration, which showed very few ions present for a weak acid, and point out that, since such a small amount is subtracted from 10^{-1} , this value is not changed significantly.) Thus

$$\frac{[\text{H}^+][\text{CH}_3\text{COO}^-]}{[\text{CH}_3\text{COOH}]} = \frac{10^{-3} \times 10^{-3}}{10^{-1}} = 10^{-5}$$

The accepted value at 25°C is 1.8×10^{-5} .

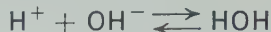
For 1 *M* acetic acid, the concentration of the H^+ corresponds to a value between that of the 0.01 *M* and the 0.001 *M* HCl. The student may use either of these and get a constant of 10^{-4} or 10^{-6} . Class discussion of these results should indicate the intermediate value of 10^{-5} .

- 2) Predict qualitatively the effect of each of the following experiments on the acetic acid equilibrium reaction:
 - a. Some of the salt sodium acetate, which produces the ions $\text{Na}^+(\text{aq})$ and $\text{CH}_3\text{COO}^-(\text{aq})$, is dissolved in 0.1 *M* acetic acid solution. Would the $[\text{H}^+]$ increase or decrease?
 - b. Some sodium hydroxide solution is added, drop by drop, to 0.1 *M* CH_3COOH solution.
 - a) The $[\text{H}^+]$ would decrease. The student may reason on the basis of a shift to favor the reactant, and this would eliminate some of the H^+ . On the other hand, the student may use the equilibrium constant just calculated.

$$\frac{[\text{H}^+][\text{CH}_3\text{COO}^-]}{[\text{CH}_3\text{COOH}]} = 10^{-5}$$

and show that any increase in $[\text{CH}_3\text{COO}^-]$ must result in an increase in $[\text{CH}_3\text{COOH}]$ and a decrease in $[\text{H}^+]$.

- b) The $[H^+]$ would decrease, causing more CH_3COOH to ionize thus increasing the $[CH_3COO^-]$. The student has seen this in Part V, and he also knows the reaction



- 3) Explain the results obtained in Part V, where you compared the number of drops of 0.1 M NaOH solution required to reach the phenolphthalein endpoint, using equal volumes of 0.1 M HCl and 0.1 M CH_3COOH solutions.

The same volume was used in each case, indicating that although there may not be as many hydrogen ions in the solution at any one time, there is the same *amount* of H^+ in equal volumes of CH_3COOH and HCl.

- 4) Calculate the $[H^+]$ of each of the solutions prepared in Part II. Use the relation

$$[H^+][OH^-] = 10^{-14}$$

In Part II the OH^- concentrations were 10^{-1} , 10^{-2} , 10^{-3} , and 10^{-4} M . The corresponding H^+ concentrations are 10^{-13} , 10^{-12} , 10^{-11} , and 10^{-10} M .

- 5) Calculate the pH of each of the solutions prepared in Parts I and II.

Solution in Part	pH
I a	1
b	2
c	3
d	4
II a	13
b	12
c	11
d	10

- 6) Determine the $[H^+]$ of your unknown solution.

See values given in Lab Hint 3.

- 7) The $H^+(aq)$ concentration of a 1 M solution of benzoic acid is 8×10^{-3} M .

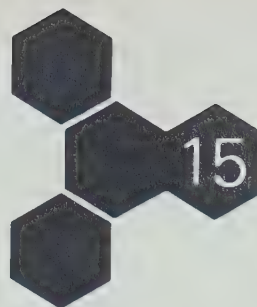
- a) What percent of the benzoic acid, C_6H_5COOH , is ionized in this aqueous solution?
 b) Predict what volume of 1 M NaOH would be required to neutralize 10 ml of 1 M benzoic acid.

- a) If 8×10^{-3} mole of H^+ is produced from one mole of benzoic acid, the percent ionized is

$$\frac{8 \times 10^{-3} \text{ mole}}{1 \text{ mole}} \times 100 = 0.8\%$$

- b) 10 ml of 1 M C_6H_5COOH would contain 0.01 mole of H^+ and would require 0.01 mole of OH^- , which would be supplied by 10 ml of 1 M NaOH.

Oxidation and Reduction



Here the ranking of half-reactions according to electron donation comes directly from an experiment (34). The student sees visual evidence of the conversion of one substance into another and relates this to the voltage produced in a cell. Experiment 35 gives a practical application to the corrosion of iron while Experiment 36 connects electrical change to mass effects. A direct confirmation of the molar relations existing in a reaction is obtained.

Schedule of Experimental Work

TEXT SECTION	EXPERIMENTAL WORK	FOR DETAILS SEE PAGE
15-1		
15-2	Expt. 34	194
15-3 to 15-7		
	Expt. 35	198
15-8, 15-9		
	Expt. 36	202
15-10 to 15-13		

Experiment 34

An Introduction to Oxidation-Reduction

PURPOSE AND SUMMARY

To establish experimentally the relative electron-losing tendency of three metals and the relative electron-gaining tendency of three halogens.

The student tests three metals (Cu, Zn, Pb) against ions of these metals. He also tests the halogens (Cl_2 , Br_2 , I_2) against their ions.

TIMING

About 50 minutes are needed during Section 15-2.

MATERIALS NEEDED

per student

12 test tubes, 13 × 100 mm

6 metal strips, about 1 × 11 cm (2 each of Cu, Zn, Pb). See Lab Hint 1.

steel wool or sandpaper

plastic thumb protectors

Solutions and reagents:

$\text{Cu}(\text{NO}_3)_2$, 0.1 M, 9 ml (24.2 g $\text{Cu}(\text{NO}_3)_2 \cdot 3 \text{H}_2\text{O}$ /liter)

$\text{Pb}(\text{NO}_3)_2$, 0.1 M, 9 ml (33.1 g $\text{Pb}(\text{NO}_3)_2$ /liter)

$\text{Zn}(\text{NO}_3)_2$, 0.1 M, 9 ml (29.8 g $\text{Zn}(\text{NO}_3)_2 \cdot 6 \text{H}_2\text{O}$ /liter)

NaBr , 0.1 M, 6 ml (10.3 g NaBr /liter)

NaI , 0.1 M, 6 ml (15 g NaI /liter)

NaCl , 0.1 M, 6 ml (5.8 g NaCl /liter)

chlorine water, saturated, 5 ml. See Lab Hint 2.

bromine water, 4 ml (1 ml Br_2 /1000 ml H_2O). See Lab Hint 3.

iodine in water-ethanol solution, 4 ml. See Lab Hint 4.

trichlorotrifluoroethane, TTE, 9 ml. See Lab Hint 5.

LAB HINTS

1. Two strips of each metal are sufficient if the student does not test the metal with the nitrate of the same element. Three pieces of each metal are needed if that test is made. The metal strips can be used many times.
2. To make saturated chlorine water, slowly pass chlorine gas from a lecture bottle of compressed gas into the needed amount of water, or set up a chlorine gas generator. Use a long-stem funnel and delivery tube in a 2-hole stopper inserted in a 250-ml flask. Add about 40 ml of concentrated HCl to about 10 g of MnO_2 in the flask, and warm gently. Use a fume hood. This recipe produces enough to prepare 1 liter of chlorine water.

3. Prepare bromine water by dissolving about 1 ml of bromine in 200 ml of water. Make the bromine by heating 10 g of KBr with 2 g of MnO_2 and 15 ml of 9 M H_2SO_4 in a glass retort and allowing the bromine to flow into a tube of water. The solution for this experiment need be only lightly colored to be concentrated enough.

CAUTION: Prepare Br_2 (l) only in a good hood. Use gloves and do not breathe the fumes. Keep a bottle of glycerol handy to rub on the skin if Br_2 (l) should inadvertently touch you.

4. Dissolve about 0.25 g of I_2 (s) in 50 ml of ethanol, and add 350 ml of water.
5. Trichlorotrifluoroethane (TTE) is very much safer than carbon tetrachloride. Since the halogens are essentially the same color in it as in CCl_4 , there is no reason to use CCl_4 . TTE costs a little less than twice as much as CCl_4 , when purchased as TF Solvent (duPont). Threshold limit values (TLV) are the amounts known to be safe for 8-hour continuous exposure. Some TLV's of interest are: CO_2 9000, TTE 7600, benzene 80, CCl_4 65 mg/m³ air.

PRELAB DISCUSSION

Demonstrate $\text{Fe(s)} + \text{Cu}^{2+}$. Have students write an equation for the reaction, assuming one product is Fe^{2+} . Discuss and label oxidizing agent, reducing agent, oxidation, and reduction. Emphasize the need to observe the extent of the reaction. A very slight discoloration of a metallic surface could be due to a very minor impurity and not to the ion being deliberately used.

PRECAUTIONS

Observe the usual lab precautions. Wear safety glasses and aprons. Treat bromine with great respect. See Lab Hint 3.

POSTLAB DISCUSSION

Be sure the student understands the idea of relative tendency to lose electrons, and how this leads to the table in Appendix 4.

SAMPLE DATA

PART I

No reaction is evident when a metal strip is left in a solution of its own ions, although Zn may react with H^+ in the $\text{Zn}(\text{NO}_3)_2$ solution. Shiny crystals of lead will be observable on the zinc strip placed in Pb^{2+} , and a copper coating will form on the zinc strip in Cu^{2+} . The lead strip placed in Cu^{2+} will also become copper coated. There will be no reaction with the copper or lead strips placed in Zn^{2+} or with the copper strip in Pb^{2+} solution.

PART II

- d) The I_2 (s) colored the TTE much more than it did the water. Presumably it is more soluble in TTE.
- e) TTE sank. Its density is 1.6 g/cm³. The color left the water and the TTE became purple.
- f) I^- caused no apparent reaction with TTE.
- g) In all cases the aqueous layer lost color. The TTE gained color and became in Cl_2 very slight yellow, in Br_2 amber, and in I_2 purple.
- h) Addition of Cl_2 caused the TTE layer in the Br^- tube to become amber. In the I^- tube it became purple.

- i) Addition of Br_2 caused the TTE layer in the Cl^- tube to become amber. In the I^- tube it became purple.
- j) In both cases, the TTE became purple.

RESULTS OF PROCESSING THE DATA

- 1) Which of the metals tested was oxidized by both of the solutions of the other metallic ions?

Zinc.

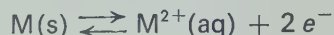
Which one was oxidized by only one of the metallic ions?

Lead.

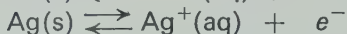
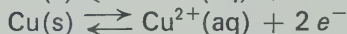
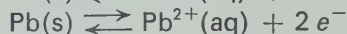
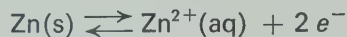
Which one was oxidized by neither of the other metallic ions?

Copper.

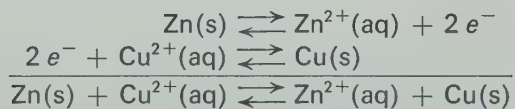
- 2) Compare the metal-metal ion pairs by arranging their half-reactions,



in a column showing order of decreasing ease of oxidation. Since you know from Experiment 7 that Cu(s) was oxidized by $\text{Ag}^+(\text{aq})$, add the $\text{Ag(s)} \rightleftharpoons \text{Ag}^+(\text{aq}) + e^-$ half-reaction to your list in the appropriate place.



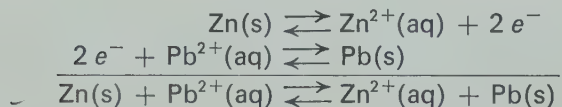
- 3) Write balanced equations for the cases where oxidation-reduction reactions between metals and metallic ions were observed [see the example for Cu(s) and $\text{Ag}^+(\text{aq})$ in the introductory section of this experiment].



oxidation half-reaction

reduction half-reaction

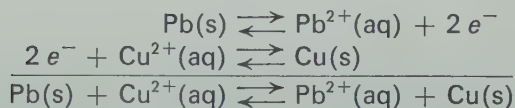
overall reaction



oxidation half-reaction

reduction half-reaction

overall reaction



oxidation half-reaction

reduction half-reaction

overall reaction

- 4) Which of the halide ions tested was oxidized by both of the other halogen elements?

Iodide.

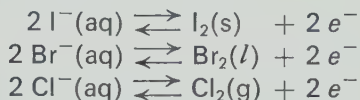
Which halide ion was oxidized by only one halogen element?

Bromide.

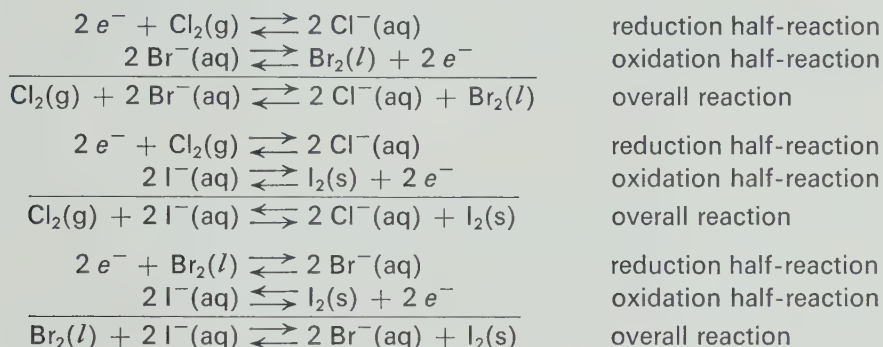
Which halide ion was not oxidized by any of the halogen elements used?

Chloride.

- 5) Compare the halide ion-halogen element pairs by arranging their half-reactions in a column in order of decreasing ease of oxidation.

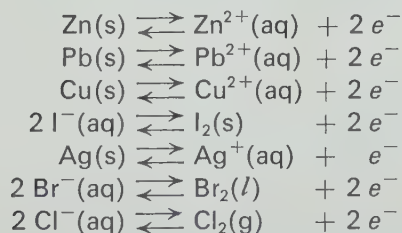


- 6) Write balanced equations for the cases where oxidation-reduction reactions occurred between halide ions and elementary halogens.



- 7) Use the additional information given below to construct a series of all seven half-reactions discussed in this experiment in order of decreasing ease of oxidation.

- a. $\text{Ag}^{+}(\text{aq})$ is a stronger oxidizing agent than $\text{I}_2(\text{s})$ but weaker than $\text{Br}_2(\text{l})$.
b. $\text{I}^{-}(\text{aq})$ is a weaker reducing agent than $\text{Cu}(\text{s})$ but is a stronger one than $\text{Ag}(\text{s})$.



- 8) Would it be feasible to store a solution of copper sulfate in a container made of metallic zinc? Of metallic silver? Give reasons for your answers.

A solution of copper sulfate would contain $\text{Cu}^{2+}(\text{aq})$ and would react with a container made of $\text{Zn}(\text{s})$, since $\text{Zn}(\text{s})$ is a stronger reducing agent (is oxidized more easily) than $\text{Cu}(\text{s})$. The CuSO_4 solution would not react with $\text{Ag}(\text{s})$, because $\text{Ag}(\text{s})$ is a weaker reducing agent (is oxidized less easily) than $\text{Cu}(\text{s})$.

Experiment 35

Corrosion of Iron

PURPOSE AND SUMMARY

To investigate some of the conditions which protect iron from corrosion and some that accelerate corrosion.

In Part I the students test the behavior of iron nails in various solutions and seek regularities in the behavior. In Part II they investigate reactions involving metal couples. Some metals, when in contact with iron in the presence of water, are found to reduce the rate of oxidation of iron; others under similar conditions seem to accelerate the oxidation of iron.

TIMING

This experiment should be done before Section 15-8 in the Textbook. It will require about $1\frac{1}{2}$ periods. Do Parts Ia through c and Parts IIe through i on the first day.

MATERIALS NEEDED

per student

- 5 test tubes, 13×100 mm
- 1 beaker, 250 ml
- 1 ring stand, iron ring, wire gauze, and burner
- 2 petri dishes. See Lab Hint 1.
- litmus paper or Hydrion paper
- distilled water (freshly boiled to reduce dissolved CO_2)
- 9 iron nails. See Lab Hint 2.
- 1 zinc strip, $2 \text{ mm} \times 10 \text{ cm}$. See Lab Hint 3.
- 10 cm copper wire, bare, 18 or 20 gauge
- 2 grams Agar, powdered
- ferrous sulfate solution, 1 ml 0.1 *M*. See Lab Hint 4.
(2.8 grams $\text{FeSO}_4 \cdot 7 \text{ H}_2\text{O}$ /100 ml of solution)
- potassium ferricyanide, 2 ml 0.1 *M*
(3.3 grams $\text{K}_3\text{Fe}(\text{CN})_6$ /100 ml of solution)
- phenolphthalein solution, 0.5 ml 1%
(1 gram/100 ml 50% ethanol solution)
- 5 ml each from one group of the following. See Lab Hint 5.

Group A

- NaOH, 0.1 *M* (0.4 gram/100 ml of solution)
- $\text{Na}_2\text{Cr}_2\text{O}_7$, 0.1 *M* (3 grams $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2 \text{ H}_2\text{O}$ /100 ml of solution)
- NaCl, 0.1 *M* (0.6 gram/100 ml of solution)
- HCl, 0.1 *M* (8.6 ml conc. reagent/100 ml of solution)

Group B

KOH,	0.1 M (0.6 gram/100 ml of solution)
Na ₂ CO ₃ ,	0.1 M (1.2 grams Na ₂ CO ₃ ·H ₂ O/100 ml of solution)
KNO ₃ ,	0.1 M (1.0 gram/100 ml of solution)
HNO ₃ ,	0.1 M (6.4 ml conc. reagent/100 ml of solution)

Group C

Na ₃ PO ₄ ,	0.1 M (3.8 grams Na ₃ PO ₄ ·12 H ₂ O/100 ml of solution)
Na ₂ C ₂ O ₄ ,	0.1 M (1.3 grams/100 ml of solution)
NaSCN,	0.1 M (0.8 gram/100 ml of solution)
H ₂ SO ₄ ,	0.1 M (2.8 ml conc. reagent/100 ml of solution)

LAB HINTS

1. Disposable plastic petri dishes are convenient, but if petri dishes are not available, use beakers.
2. The size of the nails is not important, but be sure they are *not* specially treated corrosion-resistant. Plain iron nails should be used. Be sure they are clean and free from oil or grease. Six to eight-penny size is convenient.
3. A thin zinc strip or ribbon is best, but mossy zinc can be used instead. Make sure there is good contact between the nail and the zinc.
4. The ferrous sulfate solution should be freshly prepared. Add 2 or 3 ml of concentrated sulfuric acid and an iron nail to the freshly prepared solution to maintain the iron in the +2 oxidation state.
5. Solutions may be stored conveniently in plastic squeeze bottles. Prepare three sets, but assign only one to each pair of students. Make arrangements so that class data may be obtained and shared by all students.

PRELAB DISCUSSION

None is needed.

PRECAUTIONS

Observe the usual laboratory precautions. Have students wear safety glasses and aprons.

POSTLAB DISCUSSION

Collect and discuss class data. Help the students search for regularities. There are more questions and exercises under *Processing the Data* than should be assigned at one time. Perhaps you would like to discuss some of them in class and select some to be done outside of class.

This experiment opens up an area in which there are many unanswered questions. Probably the students will not all observe the same behavior because there are a number of factors involved that are not controlled. Some of these factors are impurities in the water, gases dissolving from the air, condition of the nails, temperature, etc.

NOTE ON CORROSION

The nature of corrosion is almost always the same—a flow of electrons between certain areas of a metal surface or between different metals. This action causes metallic ions to be produced at areas that are anodes. If the metal is in water, the ions formed are carried away; the ions in the solution also aid in the transfer of electrons, increasing the corrosive activity. In the process, hydrogen is given off from the areas that are cathodes. Since hydroxide accumulates in the solution, it is not surprising that hydroxide ions already in solution might inhibit this reaction. The accumulation of hydrogen gas at the cathode (polarization) may also inhibit the reaction (this also occurs in neutral solutions). But dissolved oxygen often combines with the hydrogen, depolarizing the electrode and allowing the corrosion to proceed. Other secondary reactions also take place, such as the oxidation of ferrous hydroxide to ferric hydroxide in the corrosion of iron.

SAMPLE DATA

	Acidity as Indicated by Litmus	Rust Evident	Other Change	Result of Test for Fe^{2+}	Appearance When Dried
NaOH	basic	no	none	negative	shiny
$\text{Na}_2\text{Cr}_2\text{O}_7$	neutral	no	none	negative	shiny
NaCl	neutral	very slight	none	blue*	corrosion, especially on head
HCl	acidic	no	bubbles	deep blue	corrosion
KOH	basic	no	none	negative	shiny
Na_2CO_3	basic	no	none	negative	slightly dull
KNO_3	neutral	on bottom of tube	none	some blue on head	quite corroded, especially on head
HNO_3	acidic	no	bubbles	deep blue	corrosion
Na_3PO_4	basic	no	nail less shiny	negative	very slightly tarnished
$\text{Na}_2\text{C}_2\text{O}_4$	neutral	no	none	negative	slightly tarnished
NaSCN	neutral	on bottom of tube	none	some blue on head	very rusted
H_2SO_4	acidic	no	bubbles	deep blue	much corrosion
H_2O	slightly acidic	on bottom of tube	none	negative	considerable corrosion

* This occurred in two out of three trials.

RESULTS OF PROCESSING THE DATA

- 1) List the reagents in Part I in which no indication of corrosion was observed.

None was observed for NaOH, $\text{Na}_2\text{Cr}_2\text{O}_7$, KOH; a little was observed for Na_2CO_3 , Na_3PO_4 , $\text{Na}_2\text{C}_2\text{O}_4$.

- 2) List the reagents in Part I in which there was an indication of corrosion.

Acids (all), NaCl, KNO_3 , NaSCN, water.

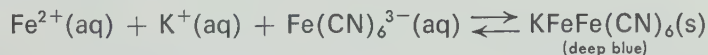
- 3) Are any regularities in evidence? What is the evidence? How do you account for the regularity?

Possible Answers: A basic condition inhibits action; acid and other ions favor it. Some students may answer "no" to the first question; tell them not to get discouraged. Point out that much variation exists between nails; in addition, other conditions are seldom controlled.

- 4) What did you observe regarding the reactions at the head, the pointed end, or at the sharp bend of the nail which were different from the rest of the nail? Account for this in terms of the mechanical treatment of the nail during its manufacture.

In the solutions in Part I, rusting was usually most evident on the head. In the agar plates, rusting was clearly most evident on the head, and sometimes also on the tip. Sometimes only the head or tip was involved; sometimes both. During manufacture the head and tip have been subjected to more stress than the shaft of the nail. Forming the sharp bend also introduces stress, and corrosion may be more evident there also.

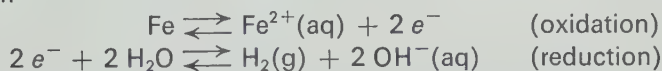
- 5) Ferrous ions react with potassium ferricyanide to form a colored precipitate. Write an equation for the reaction.



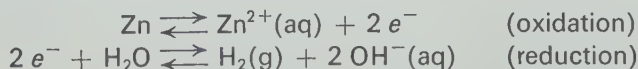
- 6) Which color in Part II indicates the site of oxidation? Which, the site of reduction? Account for the formation of each color.

The blue color (see answer to Question 5) indicates formation of Fe^{2+} , and hence oxidation. The pink color of the phenolphthalein indicates formation of OH^{-} , and hence the site of the reduction.

- 7) Write the oxidation and reduction reactions for each experiment in which you observed a reaction in Part II.



These are the reactions for the nail alone and for the nail with the copper strip. No bubbles of hydrogen will be evident because the reaction is slow. With the zinc strip the reactions are



Some solid $\text{Zn}(\text{OH})_2$ may form and show up as a white cloudiness in the agar. Zinc ferricyanide is also a white solid.

- 8) Consult the table in Appendix 4 and predict another metal that is more readily oxidized than iron and will protect it from corrosion. You may want to test your prediction by an experiment.

Any metal with a higher E° than $\text{Fe} \rightleftharpoons \text{Fe}^{2+} + 2e^{-}$ ($E^{\circ} = 0.44$ volt) can protect iron. Examples are Mg ($E^{\circ} = 2.37$) or Al ($E^{\circ} = 1.66$). The student will surely know by this time that metals such as Na and K will be too reactive to be practical and that many of the others will not be available. When tested by experiment the Mg works

well, but Al (both foil and wire were tried) does not. Probably the oxide coating prevents contact between the metals, hence a metal-metal couple is not produced.

9) How does a coating of zinc on iron (galvanized iron) protect iron from corrosion?

Zinc serves as an anode and protects the iron by keeping it reduced to Fe. The oxidation of zinc is limited, since the zinc oxide bonds tightly to the surface and serves as protection.

10) Magnesium metal rods are sometimes placed in hot water heaters. Why?

This question implies a heater having an iron tank. The Mg is oxidized more easily than Fe, hence serves as the anode. Metals are referred to as "sacrificial" when used in this way.

Experiment 36

Moles of Copper, Moles of Silver, and Moles of Electrons Involved During Electrolysis

PURPOSE AND SUMMARY

To demonstrate the quantitative relations between numbers of electrons and atoms deposited during electrolysis. Students work in groups (2-5). They plate out Ag and Cu. The amounts are determined to the nearest centigram. In processing the data, the mole concept is used extensively.

TIMING

About 55 minutes are needed before Section 15-10. The deposition of about 0.6 gram of copper and 2.0 grams of silver requires 1.0 amp for a 30-minute period. If this does not allow enough time to finish the experiment, the electrodes may be weighed the day before. They may also be left overnight after the electroplating if they are thoroughly washed.

MATERIALS NEEDED

per group

copper sulfate electrolyte solution, 400 ml. See Lab Hint 1.

silver thiosulfate electrolyte solution, 400 ml. See Lab Hint 2.

D.C. source. See Lab Hint 3.

D.C. ammeter (at least 1 amp; Welch 3031H is excellent)

variable resistor. See Lab Hint 4.

5 lengths (20-30 cm) of copper wire, 4 alligator clips. See Lab Hint 5 and diagram in Laboratory Manual.

2 pieces (6×16 cm) copper screen. See Lab Hint 6.
copper, 1 piece with wire attached. See Lab Hint 7.
lead, 1 strip with wire attached. See Lab Hint 7.
2 holders for electrodes. See Lab Hint 8.
balance, centigram
2 beakers, 400 ml

in laboratory

several clocks with second hands
acetone, 600 ml

LAB HINTS

1. Preparation of copper electrolyte solution

For 1 liter of solution :

200 g $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$

150 ml 6 *M* H_2SO_4 (or 50 ml conc. H_2SO_4)

To approximately 600 ml water, add the copper sulfate, then the sulfuric acid (take care if concentrated H_2SO_4 is used). Add water to make 1 liter. If 1 *M* copper sulfate is available, to one liter add 200 ml 6 *M* sulfuric acid.

If the electrolyte is to be used next year, add to each liter, 15 ml 6 *M* H_2SO_4 to replace the $\text{H}^+(\text{aq})$ discharged. If $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$ precipitates, add water to dissolve.

2. Preparation of silver electrolyte solution

For 1 liter of solution :

170 g $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5 \text{H}_2\text{O}$ or 108 g $\text{Na}_2\text{S}_2\text{O}_3$ (anhydrous)

22 g $\text{Na}_2\text{S}_2\text{O}_5$ (sodium metabisulfite)

50 g $\text{Na}_2\text{SO}_4 \cdot 10 \text{H}_2\text{O}$ or 22 g Na_2SO_4 (anhydrous)

25 g NaCH_3COO (anhydrous) or 41 g $\text{NaCH}_3\text{COO} \cdot 3 \text{H}_2\text{O}$

40 g AgCl

Dissolve the thiosulfate, metabisulfite, sulfate, and acetate in 1 liter of water. Dissolve the AgCl in this solution. Be sure to pulverize the AgCl and warm the solution slightly (not above 35°C) to aid in dissolving. The AgCl from Experiment 7 may be used after thorough washing. Additional AgCl may be freshly precipitated as needed, using AgNO_3 solution and NaCl solution. The AgCl must be washed before dissolving it in the hypo solution. Try to eliminate all the NO_3^- , since these ions interfere with the firm attachment of Ag plate.

To prepare 40 grams of AgCl :

Dissolve 50 g AgNO_3 in 100 ml H_2O ;

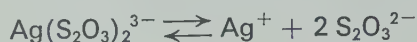
Dissolve 30 g NaCl in 100 ml H_2O ;

Mix the two solutions, heat to boiling, decant, and wash the precipitate.

The electrolyte may be replenished after each 30 minute run by adding about 12 grams of AgCl /liter. It is wise to check the *pH*. The sodium acetate will probably keep the *pH* near 5 for four runs (with Ag^+ replenished after each use), but if the solution becomes yellow and the *pH* drops to 3, add more sodium acetate and a little hypo as well as AgCl . Sometimes a small amount of milky white precipitate forms, but the cell seems to work satisfactorily.

At the lead electrode, O_2 is produced, and the lead itself is very little changed. The solution does become acidic, however, and unless a buffer is used, the thiosulfate is reduced to sulfide, which precipitates black Ag_2S .

The silver electrolyte solution contains the following ions in equilibrium:

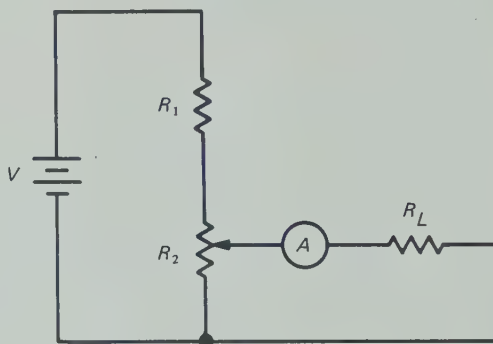


The equilibrium constant is 3.5×10^{-14} . Note the similarity in the equilibrium involved here, and in the use of this electrolyte to dissolve the unexposed $AgBr$ from the emulsion in photographic film.

3. D.C. Source

For use as a D.C. source, Heathkit "battery eliminators" are fine. Selenium rectifiers are often available at "surplus" stores. A combination of one of these with a Variac or Powerstat is good. Storage batteries, such as 12-volt automobile batteries, can also be used.

Figure 27
Electrolysis circuit
for Experiment 36.



Since current sources are not consistent, and since ammeters vary greatly, do not attempt to predict the uncertainty of measurement in this experiment, but instead collect data from as many runs as possible, and then discuss the variables.

4. Variable resistor

The size of the variable resistor used depends upon the voltage of the battery used and upon the number of electrolytic cells placed in the circuit. If a battery is available for each experimental group, the load resistance will be two or three ohms. A suitable circuit is shown in Figure 27. If the sum of the resistances for the two cells in series (R_L) is 2 or 3 ohms, the variable resistance, R_2 , should be 18–23 ohms. The size of the auxiliary resistance, R_1 , depends upon the voltage. The table below shows how large R_1 should be in order to pass 1.0 amp through the cell and draw 1.2 amps from the battery. Notice that R_1 and R_2 must be able to carry a current of 1.2 amps continuously.

Values for R_1 for Various Experimental Conditions

Battery voltage (volts)	R_1 (ohms) needed if $R_2 = 18$ ohms	R_1 (ohms) needed if $R_2 = 23$ ohms
6	0	—
12	5	0
24	15	10
100	80	75
120	95	90

CAUTION: If your only source of D.C. voltage is 100 or 120 volts, fuse the circuit for 2 amps, and take suitable precautions to avoid electrical shorts and shocks.

5. Copper Wire

Copper bell wire is suitable (preferably insulated). Alligator clips should be provided for connections.

6. Cathodes

A good grade copper window screen is satisfactory, but a finer mesh is better. Most scientific supply companies carry 20 mesh wire gauze, which has eight copper wires per centimeter. A piece 8×22 cm has about 340 cm^2 surface, which, with a current of 1 amp, gives an adequate current density for Cu, and has been found to be satisfactory for the Ag plating in this experiment. See Figure 36-1, p. 92 in the Lab Manual.

Prepare the cathodes beforehand. They may be used many times. Loose pieces of wire should be removed after cutting so that they will not fall off between weighings. Make a cylinder of the screen by weaving copper wire through about 1 cm of overlap.

The screens may be cut and fashioned into cylinders at any time, but the following cleaning process should be performed just before the experiment (the day before, but not much sooner). The first time the screen is used, clean it by using "bright dip" cleaning solution made as follows:

Add slowly to 490 ml of water
435 ml conc. (18 M) H_2SO_4

Then add: 72 ml conc. (16 M) HNO_3
3 ml conc. (12 M) HCl

This must be used in a hood. Dip the screen briefly (up to 5 seconds), and quickly rinse with water. If the surface is not bright copper, dip again. As soon as washed, dip in acetone, and allow to dry. Do not use this dip for the lead anodes.

The screen that is to be used for the silver plating electrode should be given a thin coat of silver. To do this, hook one up in a circuit as in Lab Manual Figure 36-1. Use a 600-ml beaker. Adjust the resistor for maximum resistance. Pour silver electrolyte into the beaker to cover the electrode. Adjust for a 0.1 ampere current. Operate only a few minutes until the copper is covered with a thin layer of silver. Wash it first with water, then with acetone, and allow it to dry.

It is useful to have the cathodes numbered. This facilitates following a particular electrode from experiment to experiment, thereby affording a check on the students' mass determination. Cut 1×1 cm squares of sheet Cu. Stamp a number on each. Solder one to the top of each electrode.

7. Anodes

Cut copper sheet into 2.5×17 cm strips. Center a 12-cm piece of about 20-gauge copper wire across the short center of the strip. Solder it and bend the strip double so the solder is inside.

This large area reduces voltage drop due to polarization. If a coil of wire is used, 160 cm of 16 gauge is needed to produce the same area as the above strip.

A strip of sheet lead $2\frac{1}{2} \times 8$ cm is suitable. If sheet lead is not available, use several thicknesses of lead foil. Attach a 12 cm Cu wire by crimping and soldering it near the top.

8. The electrodes need to be held in place so that they will not touch each other. A cross made from $\frac{1}{8}$ " Masonite is useful. Glue two 9.5×3 cm pieces to make a symmetrical right angled cross. Drill two small holes 1.3 cm on either side of center to take the wires from the central anode. Drill two small holes 2.6 cm on either side of center and at right angles to the first pair. The last pair of holes will take the wires from the wire screen cathodes.

Contact between electrodes can also be prevented by housing the anodes in a cylinder made of plastic window screen.

9. After several platings the silver can be reclaimed if you desire. Some silver will flake off if the screen is twisted back and forth and/or brushed with a stiff brush. Dissolve the silver flakes in nitric acid. Nitric acid may also be used directly on the screen, but considerable copper will also dissolve. Since it is difficult to preserve the screen during such treatment, we suggest you use new ones for the next year. The silver nitrate produced in this manner may be used to precipitate AgCl for next year's experiment. The AgNO₃ will probably contain too much copper to be suitable for Experiment 7. See *J. Chem. Education*, **36**, 286–287 (1959) for additional details on separating Ag from Cu.

PRELAB DISCUSSION

Although the actual ratio of silver to copper has been previously determined (Experiment 7) and the ratio of electrons to silver and copper ions previously studied (Chapters 6 and 9), this experiment will remain a challenge to most students because of the precision possible and the complexity of equipment. Point out that the focus of interest in this experiment has to do with the method rather than with the quantitative analysis of the data. Explain that getting a successful plate on a metal surface is dependent upon many factors—the cleanliness of the surface to be plated, the voltage, the current density (ratio of amps to surface area), the source of the ion to be deposited. In this experiment the copper anode supplies the copper ions, which are then plated on the cathode. For review define the cathode as the electrode at which reduction occurs; the anode as the electrode at which oxidation occurs (from Chapter 15).

The Cu²⁺(aq) is continually replaced; thus the concentration does not change during the process. In contrast to this, the silver that is plated comes from the solution, where it is present as a complex ion. There is no need to go into details here.

Remind students to have all connections tight and to check the wiring carefully. Do not rub or twist the cathodes while removing or drying them. Some metal may flake off.

Point out that there is negligible danger of electrical shock from 12 volts.

There may be a drop in current after a few minutes, probably due to polarization. Stirring or placing the beakers on hot plates helps prevent this drop. However, the current usually becomes quite steady as the reaction proceeds and frequently needs little adjusting after the first few minutes.

PRECAUTIONS

The CuSO₄ solution contains H₂SO₄. Although it is dilute, take care to avoid dripping when electrodes are withdrawn. The silver electrolyte will cause stains on hands and clothing if not rinsed off immediately after contact. Wear safety glasses and aprons.

POSTLAB DISCUSSION

Focus the discussion of the circuit on the fact that the current is the same in all parts of the circuit. The metallic copper and silver are plated by the same current.

The ratio of Cu/Ag plated will be more reliable than the ratio of either to electrons. Collect class data, and discuss the possible errors and the uncertainty in the measurements. Be sure that your students realize the significance this experiment holds with regard to our knowledge of the composition of matter.

SAMPLE DATA

Copper Cathode in Copper Sulfate Electrolyte		Copper Cathode in Silver-hypo Electrolyte	
Original mass	8.26 ± 0.01 g	Original mass	8.41 ± 0.01 g
Final mass	8.86 ± 0.01 g	Final mass	10.44 ± 0.01 g
Cu deposited	0.60 ± 0.02 g	Ag deposited	2.03 ± 0.02 g
<i>Anode (optional)</i>			
Original mass	6.26 ± 0.01 g	Current	1.0 ± 0.05 amp (5%)
Final mass	5.63 ± 0.01 g	Time	30 ± 0.17 min (0.3%)
Cu removed	0.63 ± 0.02 g		(± 10 sec)

PROCESSING THE DATA

- 1) Calculate the number of moles of electrons which were used. Recall that 1.04×10^{-5} mole of electrons is involved when a current of one ampere flows for one second.

$$(1.0 \text{ amp})(30 \text{ min})(60 \text{ sec/min})(1.04 \times 10^{-5} \text{ mole of electrons/amp-sec}) \\ = 1.9 \times 10^{-2} \text{ mole of electrons}$$

The 1.04×10^{-5} mole/amp-sec is calculated from the value of the coulomb, which has an uncertainty of about $\pm 0.001\%$ —so small that it can be ignored in this calculation. A coulomb is the amount of electricity needed to deposit 1.04×10^{-5} mole of silver.

- 2) Calculate the number of moles of copper deposited on the cathode from the solution containing copper ions.

$$\frac{0.60 \text{ g Cu deposited}}{63.5 \text{ g/mole}} = 0.94 \times 10^{-2} \text{ mole Cu}$$

- 3) Calculate the number of moles of silver deposited on the cathode from the solution containing silver ions.

$$\frac{2.03 \text{ g Ag deposited}}{108 \text{ g/mole}} = 1.88 \times 10^{-2} \text{ mole Ag}$$

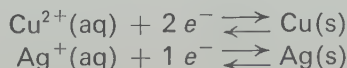
- 4) Calculate the ratio between moles of electrons and moles of copper formed. Use your answers to Questions 1 and 2.

$$\frac{\text{moles electrons}}{\text{moles Cu formed}} = \frac{1.9 \times 10^{-2} \text{ mole of electrons}}{0.94 \times 10^{-2} \text{ mole Cu}} = 2.0 \frac{\text{moles } e^-}{\text{mole Cu}}$$

- 5) Calculate the ratio between moles of electrons and moles of silver formed. Use your answers to Questions 1 and 3.

$$\frac{\text{moles electrons}}{\text{moles Ag formed}} = \frac{1.9 \times 10^{-2} \text{ mole of electrons}}{1.88 \times 10^{-2} \text{ mole of silver}} = 1.0 \frac{\text{mole } e^-}{\text{mole Ag}}$$

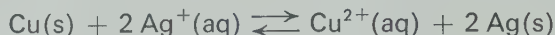
- 6) Write equations for the cathode reactions. Assume that the reacting species are simple copper ions or silver ions.



- 7) Calculate the ratio between moles of silver formed and moles of copper formed. Use your answers to Questions 2 and 3.

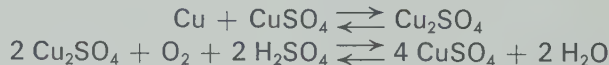
$$\frac{\text{moles of Ag formed}}{\text{moles of Cu formed}} = \frac{1.88 \times 10^{-2} \text{ mole Ag}}{0.94 \times 10^{-2} \text{ mole Cu}} = 2.0 \frac{\text{moles Ag}}{\text{mole Cu}}$$

- 8) How does this ratio compare to the ratio you obtained in Experiment 7?
It is the same.



- 9) Write the balanced equation for the oxidation reaction which occurs at the copper anode. How would you expect the loss of mass at the copper anode to compare with the gain of mass at the copper cathode?

One would expect that the two would be equal. This is the appropriate student answer. In the sample data, more mass was lost at the anode than was gained at the cathode. Such a difference, which is just barely significant, is likely to be found. It may occur because the high current density at the small-area anode causes uneven removal of the metal. Flecks of copper are often visible in the acetone when this electrode is rinsed prior to weighing. Moreover, the concentration of CuSO_4 tends to increase, and that of H_2SO_4 to decrease. Copper may exist as both CuSO_4 and Cu_2SO_4 in the sulfate solution. Copper is oxidized either to Cu^+ or Cu^{2+} at the anode. The Cu^+ is then oxidized to Cu^{2+} in solution. In addition, some Cu dissolves according to the reactions

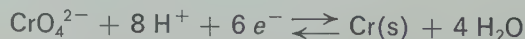


These reactions account for the observed difference in mass lost at the Cu anode compared to the mass gained at the cathode.

- 10) What are some sources of error in your measurements and procedures? How many significant figures in the mole relations can be justified by your data?

The largest error is in the measurement of the current. Not only is it difficult to hold constant, but it is uncertain to at least $\pm 5\%$. The mass of copper, about 0.60 gram, is uncertain by about ± 0.02 gram, hence introduces an uncertainty of about 3%. The uncertainty of the time measurement is negligible if measured to the nearest second. The uncertainty of 5% justifies two significant figures.

- 11) How many moles of electrons would be required to plate 52.0 grams of chromium on a cathode from a suitable cell containing an electrolyte solution in which the chromium has an oxidation number of +6?



$$\frac{52.0 \text{ g Cr}}{52.0 \text{ g/mole}} = 1.00 \text{ mole Cr}$$

Since each atom of chromium deposited requires six electrons, 1.00 mole of chromium plated requires 6.00 moles of electrons.

Molecular Structure



This chapter discusses subjects which cannot be presented in an experimental way at the high school level. Infrared and nuclear magnetic resonance spectrometers are too expensive and their use too complicated. However, the student can understand their general use. In the laboratory he pursues the use of E° tables for predicting reactions. Thus, his understanding of descriptive chemistry is broadened on a firm base of principles.

Schedule of Experimental Work

TEXT SECTION	EXPERIMENTAL WORK	FOR DETAILS SEE PAGE
16-1 to 16-7	Expt. 37*	210

* The experiment is not closely connected to this chapter. It may be done any time.

Experiment 37

Reactions Between Ions in Solution

PURPOSE AND SUMMARY

This experiment makes use of the E° table, the relative strength of acids table, and the solubility rules in order to predict results of mixing various chemicals.

In Part I the students are required to predict whether an appreciable reaction can be expected for ten different combinations of solutions. After writing balanced equations for the expected reactions, they do the experiments to find out if their predictions were correct. In Part II they are not required to predict the reactions before doing the experiments. After making observations, the student is required to deduce the reactions and write net ionic equations for them.

TIMING

This experiment can be done anytime after Chapter 15. It is not directly associated with the material of Chapter 16; however, it does serve as good summary and review of the previous three chapters.

Part I can be completed in about 35 minutes if the predictions are made prior to class time. Part II is optional and requires an additional period.

MATERIALS NEEDED

per student or pair

2 or more 13 × 100 mm test tubes
plastic thumb protectors (to be used in place of stoppers)
1 test tube rack

per class of 30 students. See Lab Hint 1.

PART I

magnesium nitrate, 200 ml 0.1 *M*

[25.6 g $\text{Mg}(\text{NO}_3)_2 \cdot 6 \text{H}_2\text{O}$ /liter]

sodium hydroxide, 100 ml 0.1 *M* (4 g/liter)

sodium sulfate, 100 ml 0.1 *M*

(14.2 g anhydrous/liter)

barium hydroxide (saturated), 200 ml 0.1 *M*

[19 g $\text{Ba}(\text{OH})_2 \cdot \text{H}_2\text{O}$ /liter or

32 g $\text{Ba}(\text{OH})_2 \cdot 8 \text{H}_2\text{O}$ /liter]

sulfuric acid, 100 ml 0.1 *M*

(5.6 ml conc. reagent/liter)

sulfuric acid, 2 ml 6 *M*

(334 ml conc. reagent/liter)

potassium dichromate, 100 ml 0.1 *M*

(29.4 g/liter)

sodium sulfite, 100 ml 0.1 *M*. (Must be fresh.

Test as in Step d of experiment.)

(12.6 g anhydrous/liter)

potassium permanganate, 100 ml 0.05 *M*

(8 g/liter)

potassium permanganate, 100 ml 0.01 *M*

(1.6 g/liter)

hydrochloric acid, 100 ml 1.0 *M*

(85.5 ml conc. reagent/liter)

potassium iodide, 100 ml 0.1 *M* (16.6 g/liter)

ferric chloride, 200 ml 0.1 *M*

(27 g $\text{FeCl}_3 \cdot 6 \text{H}_2\text{O}$ /liter)

potassium bromide, 100 ml 0.1 *M* (12 g/liter)

ferrous sulfate, acidified, 100 ml 0.1 *M*.

(27.8 g $\text{FeSO}_4 \cdot 7 \text{H}_2\text{O}$ /liter 0.01 M H_2SO_4)
(Discard excess. Do not save.)
zinc sulfate, 200 ml 0.1 M
(29 g $\text{ZnSO}_4 \cdot 7 \text{H}_2\text{O}$ /liter)

ammonium carbonate, 100 ml 0.1 M
(10 g/liter)
60 ml trichlorotrifluoroethane (TTE)

PART II (Optional)

chromium sulfate, 200 ml 0.1 M
(60 g commercial $\text{Cr}_2(\text{SO}_4)_3 \cdot n \text{H}_2\text{O}$ /liter)
sulfuric acid, 10 ml 6 M (334 ml conc.
reagent/liter), also used in Part I
hydrogen peroxide, 400 ml 3%
sodium hydroxide, 10 ml 6 M (240 g/liter)
potassium dichromate, 200 ml 0.1 M
(29.4 g/liter), also used in Part I

lead nitrate, 100 ml 0.1 M (33 g/liter)
sodium chloride, 100 ml 0.1 M (5.9 g/liter)
potassium iodide, 100 ml 0.1 M
(16.6 g/liter), also used in Part I
sodium sulfide, 100 ml 0.1 M
(24 g $\text{Na}_2\text{S} \cdot 9 \text{H}_2\text{O}$ /liter)

LAB HINTS

1. It is essential that solutions be readily available. It will help to have on hand small trays, such as inexpensive plastic pans or boxes (approximately 6×10 inches) in which reagents for a group of parts (for example, a, b, and c) may be placed. To save time and avoid all needing the same reagents at the same time, you might direct students to start at different points in the experiment. There are four natural groupings: a, b, and c; d, e, and f; g and h; and i plus j.
2. Another possible way to organize for this experiment is to have the students work on some of the predictions, discuss these in class, and then try them during a 15–20 minute period on each of several days. In this way all of the reagents will not have to be out at the same time.

PRELAB DISCUSSION

Emphasize the element of *prediction*. See that students come to the laboratory with the equations written. If they seem uncertain about predicting, suggest that they list the species present in the solution and then check the table of E° values for possible oxidation-reduction reactions and the solubility charts for possible formation of precipitates. Try to get the students to work these out entirely on their own. Remind the students that the E° tables used for predictions are strictly true only for 1 M solutions. But since the effect of concentration is not great for these reactions, the tables provide a good *approximation*.

PRECAUTIONS

Observe the usual laboratory precautions. Have the students wear safety glasses and aprons. ~

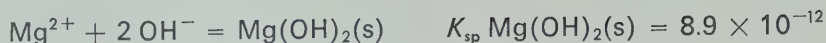
POSTLAB DISCUSSION

Discuss reactions as needed for Part I. If Part II is done by many students, a more extensive discussion of reactions will probably be needed.

Equations and Discussion of Reactions

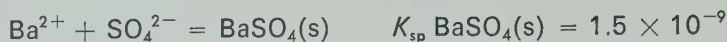
PART I

a. Precipitate predicted:



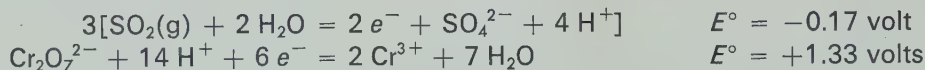
b. No precipitate is predicted. Both MgSO_4 and NaCl are quite soluble. The student may remember the low solubility of BaSO_4 and SrSO_4 if these were used in Experiment 17, but the solubility of MgSO_4 is much greater.

c. Precipitate predicted:



The student should recognize that the equilibrium $\text{H}^{+}(\text{aq}) + \text{OH}^{-}(\text{aq}) = \text{H}_2\text{O}$ also favors the products of this reaction.

d. Oxidation-reduction is predicted. The student may need to be reminded of reaction II g in Experiment 24 in which $\text{H}^{+} + \text{SO}_3^{2-}$ produced $\text{SO}_2(\text{g})$.

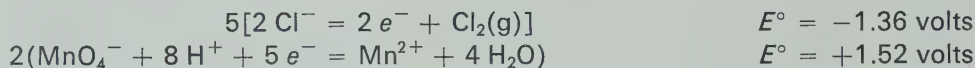


or



The color will change from orange to green. The student saw this change of $\text{Cr}_2\text{O}_7^{2-}$ to Cr^{3+} in Experiment 30.

e. Oxidation-reduction predicted:

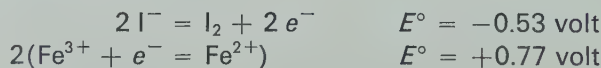


or



In the introduction to the experiment, the student has been reminded that E° tables do not predict the rate of the reaction and has been told to make observations later if no reaction is apparent immediately. This reaction will appear inactive at first, but after 5 or 10 minutes there will be no doubt that the odor of $\text{Cl}_2(\text{g})$ is present, and the pink color of the MnO_4^{-} should disappear.

f. Oxidation-reduction predicted:

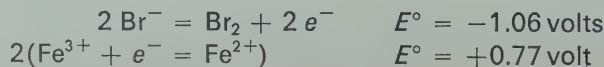


or



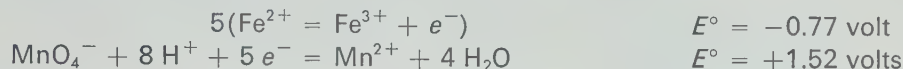
It is difficult to detect the iodine in the presence of the yellow-brown Fe^{3+} solution. But when TTE is added it extracts I_2 and becomes lavender-pink. The student is familiar with this from Experiment 34.

g. No oxidation-reduction reaction predicted:



Since the reactants are favored, the student will see no change in the color of the TTE layer.

h. Oxidation-reduction predicted :

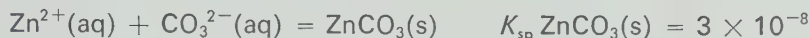


or



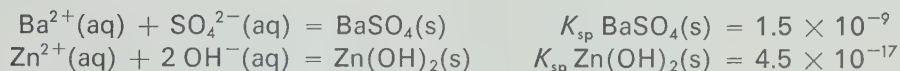
The solution will change from the purple-pink of MnO_4^- to the brown-yellow of Fe^{3+} .

i. Precipitate predicted :



This K_{sp} is not given in the Textbook, but the solubility tables will lead the student to this prediction.

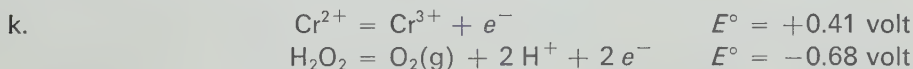
j. Precipitates predicted :



The student who is familiar with the first of these from (c) may fail to predict the second. It may be difficult to be sure whether there are two precipitates here, but most students will see a difference from the reaction in (c).

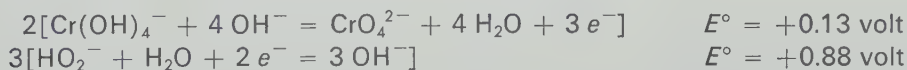
PART II (OPTIONAL)

These reactions are more involved than those of Part I and are presented with the direction that the student try them first and then seek an explanation. For several reactions the first guess (we assume the expected familiarity with the solubility and oxidation-reduction tables) will be incorrect, and a more careful consideration will be necessary. The less-talented students will not be able to reason through some of these, and even the better students may need helpful hints. There is ample opportunity for commenting that the first prediction is not always correct and that the final answer must be obtained from the laboratory.

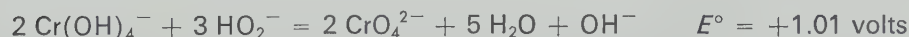


No reaction occurs.

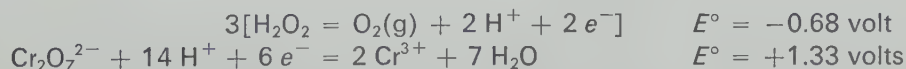
l. The color changes from green to yellow.



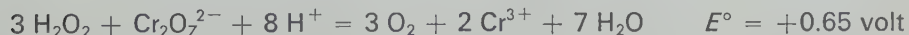
or



m. The color changes from orange to green, and bubbling occurs.



or

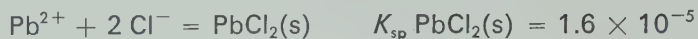


- n. No color change takes place except the orange-to-yellow change indicated in the directions. From his experience with Experiment 30 the student should remember the equilibrium reaction



The Cr^{6+} is not reduced in a basic solution.

- o. A white precipitate appears:



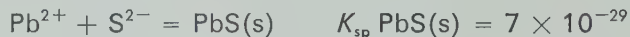
Since an excess of Cl^- was added, the Pb^{2+} remaining in solution is about $4 \times 10^{-6} \text{ M}$.

- p. A yellow precipitate appears.



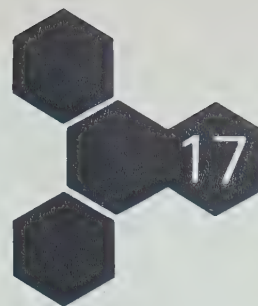
The remaining Pb^{2+} from (o) was sufficient to give a visible amount of $\text{PbI}_2(\text{s})$, since the K_{sp} for $\text{PbI}_2(\text{s})$ is smaller than that of $\text{PbCl}_2(\text{s})$.

- q. A small amount of black precipitate appears.



There were still enough Pb^{2+} ions left in the saturated PbI_2 solution to produce a small amount of $\text{PbS}(\text{s})$, since the K_{sp} for this salt is so much smaller than that for $\text{PbI}_2(\text{s})$.

Chemical Bonding in Gases, Liquids, and Solids



This chapter continues with material that is not easy to show experimentally. The orderly arrangements in solids help explain their properties and why metals are so similar. The experiment and demonstrations lead the student to see the three-dimensional relations.

Schedule of Experimental Work

TEXT SECTION	EXPERIMENTAL WORK	FOR DETAILS SEE PAGE
17-1 to 17-5		
	Expt. 38	216
	Demo. 34	220
	Demo. 35	223
17-6 to 17-14		

Experiment 38

The Packing of Atoms in Crystals

PURPOSE AND SUMMARY

To study the ways in which spheres can be packed and to relate these arrangements to common crystal structures found for metallic and ionic solids.

The student uses Styrofoam spheres to make the following structures: face-centered and hexagonal closest packed, body-centered cubic, and NaCl. He also investigates the relation between face-centered and hexagonal closest packed.

TIMING

Before Section 17-6, about 45 minutes are needed. You may choose to do Parts I–V on packing spheres of uniform size before Section 17-6 (35 minutes) and do Part VI using two sizes of spheres before Section 17-10 (10 minutes).

MATERIALS NEEDED

per pair of students

a box containing the following Styrofoam spheres:

36 2-inch spheres (one of these should be colored). See Lab Hint 1.

13 1-inch spheres

38 chenille stems, 1 inch long. See Lab Hint 2.

LAB HINTS

1. With a small amount of care, one set of spheres will serve many years. Keep the spheres off the floor and tolerate no horseplay.
2. Chenille stems are superior to pipestem cleaners as they have a longer nap. This makes a smaller hole but still provides good bite. They are obtainable from 5-and-10 cent stores and may be cut with pliers or tin snips.
3. Some students find these ideas extremely easy to grasp. Many, however, will find them difficult. Plan on being quickly available for those who have difficulty. Dividing the experiment into two parts not only makes for better correspondence with the Textbook, but also enables the student to review the packing of spheres of the same size after he has read more about the structure of solids and before he goes on with a study of ionic crystals.

PRELAB DISCUSSION

Students might be tempted to throw or crush the spheres. A few words against this are in order. It is well to have students work in pairs. This promotes discussion of questions asked in the *Procedure Section*, and provides an extra pair of hands to hold some of the spheres.

PRECAUTIONS

Do not let the spheres roll on the floor and underfoot.

POSTLAB DISCUSSION

Many students have difficulty with the subtle differences between face-centered and hexagonal closest packing. Demonstrations 34 and 35 help clarify the difference.

PARTS I-IV

Discussion of the experiment should emphasize the idea that "closest packing" is the packing of spheres of uniform size to occupy the smallest volume, hence to bring the atoms as close together as permitted by the repulsive interactions that give the atoms size. In Part I, the student learns that within one plane, the closest packing of spheres of the same size gives each sphere six nearest neighbors, no matter how large the spheres. In Part II, this packing is extended to three dimensions by using the hexagonal closest packing arrangement. Then in Parts III and IV, the cubic closest packing is demonstrated in two ways, in Part III to display the cubic arrangement, and in Part IV to contrast hexagonal and cubic closest packing. Point out that twelve nearest neighbors result from either closest-packing arrangement.

PART V

The body-centered cubic packing is not a closest packed arrangement. Each atom has only eight nearest neighbors, and the density is lower than it is in the closest-packing arrangements.

PART VI

All of the alkali halides have the NaCl-type lattice except three, CsCl, CsBr, and CsI. Oxides and sulfides of alkaline earths, as well as many other monoxides and monosulfides, some carbides, some nitrides, and a few alloys have the NaCl structure.

RESULTS OF PROCESSING THE DATA

- 1) Write a brief description of each type of packing of metallic crystals that you studied.

The student's description should include hexagonal closest packing, cubic closest packing, and body-centered cubic packing.

- 2) Answer all questions raised in the *Procedure* sections. Label them by Parts and Sections, for example, P I-a.

PART I

- (a) The maximum number of spheres that can be packed around a single sphere in the same plane is six. The number is independent of the size of the spheres if they are all the same size.
- (b) The marked sphere has twelve neighbors touching it. Since these neighbors are all at the same distance and closer than any others that can now be added, they are called *nearest neighbors*. The number of nearest neighbors is equal to the coordination number.

PART II

- (f) The coordination number is twelve.

PART III

- (h) The model is called face-centered cubic because the unit cell cube has a sphere placed in the center of each cube face.

PART IV

- (k) No. The coordination number is 12 for each type of closest packing. There is no difference in density when spheres of the same size and mass are involved.

Since most metals are found in closest packing arrangements, closeness of approach rather than fixed direction of bonding dominates the crystal structure. Nevertheless, the fact that some metals are found in cubic packing, whereas others crystallize in hexagonal packing could be interpreted to mean that some metals have a directionality that favors one closest packing and that other metals have a directionality that favors the other. A relatively small amount of directionality may be the factor that determines which of the closest-packed structures is the more stable. Yet the difference between hexagonal and cubic closest packing could also be attributed to interactions between the first and third layers of atoms rather than to nearest neighbors.

PART V

- (m) The name fits the structure in that the unit cell consists of a cube with spheres at each corner and one in the center.

The alkali metals have many vacant valence orbitals but very few valence electrons. Presumably the shortage of bonding electrons accounts for the fact that the alkali metals are more stable when the arrangement is such that each atom shares valence electrons with only eight nearest neighbors (as in body-centered cubic packing) rather than with twelve nearest neighbors (as in the closest-packed structures).

- (n) α -ferrite (alpha-ferrite) has coordination number 8.
 γ -ferrite (gamma-ferrite) has coordination number 12.
 δ -ferrite (delta-ferrite) has coordination number 8.

The effect of raising the temperature could be to excite more and more electrons to act as valence electrons. At low temperatures, perhaps there is a shortage of valence electrons, making the structure having coordination number eight the most stable. As more electrons become mobile, the structure having coordination number twelve becomes the most stable. Explaining the high-temperature transition to the δ form is more difficult. Perhaps the thermal excitation of lattice modes finally favors a less densely packed crystal that gives each atom more space in which to vibrate. The student is asked to "suggest" a reason, so detail is not to be expected. The actual answer is very complicated, having to do with how entropy and energy change with temperature.

PART VI

- (q) Surrounding each Na^+ ion are six Cl^- ions, and around each Cl^- ion are six Na^+ ions. The coordination number for each kind of ion is six. Coordination number in ionic solids is determined as for metallic structures, by counting the number of nearest neighbors. In ionic solids, the nearest neighbors have a charge opposite to that of the central atom.

$$(r) \ 0.95 \text{ \AA} / 1.81 \text{ \AA} = 1/1.91 = 0.525$$

The stability of this type of packing can be attributed to the proximity of ions of opposite charge.

- 3) In one of the types of cubic packing, the spheres occupy about two-thirds of the space, and in the other they fill about three-fourths of the space available. Identify which type is which. Which is more dense? In which does each atom form the larger number of bonds?

In the body-centered cubic packing, about two-thirds of the space is occupied by the spheres. Three-fourths of the space available is occupied by the spheres in cubic closest packing. Cubic closest packing is the more dense of the two, and has the larger number of bonds per atom.

- 4) Suppose you have a crystal XY with the sodium chloride packing in which each of the ions is the same size as the Na^+ and Cl^- respectively, but each is doubly charged X^{2+} and Y^{2-} . Would XY have a higher or lower melting temperature than NaCl ? Suggest a real pair of crystals which meets the above criteria and look up their melting temperatures to check your prediction.

Crystal XY would have a higher melting temperature, due to the stronger attractions between the ionic charges (for example, NaCl , m.t. = 801°C ; CaO , m.t. = 2850°C).

Of course, not all $X^{2+}Y^{2-}$ crystals have the NaCl structure (e.g., ZnS). The student can find tables that list the crystal structures in most handbooks under the index entry "X-ray crystallographic data"; for example, in the *Handbook of Chemistry and Physics*, 43rd ed., such a table is found on pp. 2734–2787. Some examples of NaCl -type crystals are MgO , MgS , CaO , CaS , SrO , SrS , BaO , BaS , MnO , FeO , PbS .

- 5) Suppose you have a crystal AB with the sodium chloride packing in which each of the ions has the same charge A^+ and B^- , as Na^+ and Cl^- , but the radii of A and B are proportionately larger. Would AB have a higher or lower melting temperature than NaCl ? Suggest a real pair of crystals which meets the above criteria, and look up their melting temperatures to check your prediction.

The larger the ions, the farther the charges are separated, hence the weaker will be the crystal. It would be desirable to compare salts with constant radius ratio. This isn't possible, but the trend is nevertheless detectable in the following data.

Salt	Radii, Å		Radius X^-	m.t.
	M^+	X^-	Radius M^+	
LiF	0.60	1.36	2.27	870°C
NaCl	0.95	1.81	1.91	801°C
KBr	1.33	1.95	1.47	730°C
KI	1.33	2.16	1.62	723°C

DEMONSTRATION 34
COMPARISON OF
FACE-CENTERED AND HEXAGONAL CLOSEST PACKING

PURPOSE

To reinforce and extend the comparison between face-centered closest packing and hexagonal closest packing. The student attempted this in Part IV, Experiment 38. Face-centered closest packing is also referred to as face-centered cubic packing.

TIMING

About 15 minutes are needed after the students have done Part IV of Experiment 38.

MATERIALS NEEDED

- 18 Styrofoam spheres, 3 inch diameter. See Lab Hints 1 and 3.
- 11 wood dowels, $\frac{3}{16}$ inch diameter, 7 cm long.
- 1 wood dowel, $\frac{3}{16}$ inch diameter, 18 cm long. See Lab Hint 2.
- 8 chenille stems, $1\frac{1}{2}$ inch long

LAB HINTS

1. The spheres should be rolled to the largest possible uniform diameter. Coat them with a latex paint. They can be left the color of the latex, or covered with aluminum paint to suggest a metal. Many aluminum paints and enamels attack the Styrofoam, hence the protective first coating of latex.
2. Although $\frac{1}{8}$ inch wood dowels will do, the $\frac{3}{16}$ inch dowel makes a sturdier connection. This is especially needed in the section containing five spheres.
3. Assemble two separate trios, each of which is held together with two 7 cm dowels and a chenille stem. See Figure 28. Punch holes for dowels at points of contact, put a drop of white vinyl glue in each hole, insert dowel or chenille stem, and assemble.

Assemble five spheres in a face-centered square. See Figure 29.

The last seven spheres are joined to make a cluster of six around one. See Figure 30. The following sequence facilitates assembly.

Join 1 to 2

Join 2 to 3

Join 3 to 1

Join 4 to 1 and 3 simultaneously

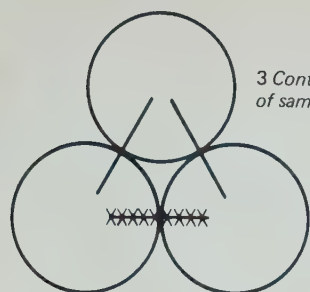
Join 5 to 4 and 1 simultaneously

Join 6 to 1 and 5 simultaneously

Join 7 to 6 and 1 simultaneously

Glue 7 to 2.

Let the glue set while the cluster rests under a weighted flat board.



3 Contacting spheres
of same size

— wood dowel

xxxxxxx chenille stem

Figure 28

Two such trios of
contacting spheres are needed.

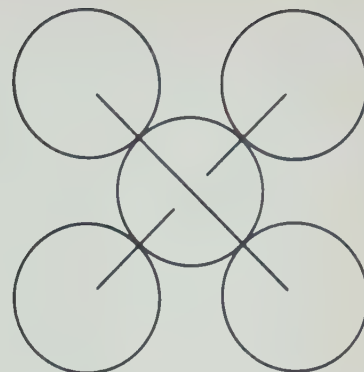


Figure 29

A 5-sphere
face-centered square.

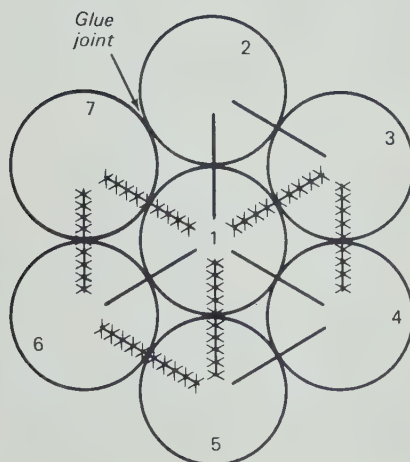


Figure 30

A closest packed cluster.

CLASSROOM PRESENTATION

Action

Arrange the trios centered and on opposite sides of the central sphere in the cluster of seven. Each sphere in one trio should be directly above a sphere in the other trio.

Rotate one trio 60°.

Comment

Is this an example of closest packing? (yes)
What is the Coordination Number (C.N.)? (12)

Is this still an example of closest packing? (yes)
What is the C.N.? (12)

Are these two arrangements really different? (yes)
How do they differ?

Show this.

First Arrangement. Each sphere in one trio is directly over a sphere in the other trio. The sequence of layers can be described as 121212 . . .

The different numbers designate closest packed layers with different horizontal displacement. All spheres in layers designated by number 1 form vertical columns of spheres. All spheres in layers designated by number 2 also form vertical columns of spheres, but these columns occur to the left or right of the first set. *Second Arrangement.* The spheres in one trio are not over spheres in the other trio.

Show this.

The sequence of layers can be described as 123123 . . .

One of these is face-centered closest packing, the other is hexagonal closest packing. Which is which? How can we find out? (We look for regularities.)

Let the students point out the 6 arrangements of 4 spheres in a square—as in the center layer of a face-centered cubic unit cell.

Perhaps we can find a face-centered cubic unit cell in this arrangement.

As an aid, position the 5-sphere unit down on one of the squares of 4 spheres. Have the two trios and cluster in the 123123 . . . sequence when you do this.

A face-centered cubic unit cell is apparent. The 123123 . . . sequence of closest packed layers is the face-centered closest packed arrangement.

Now what about the 121212 . . . arrangement?

Show it also has 6 sets of 4 spheres arranged in a square.

Is this not also face-centered closest packed? (No, a face-centered cube cannot be located.)

Use the 5-sphere unit to test this question. Show three regular face-centered squares—the one you added and two more. But the other 3 faces are not face-centered squares.

FOLLOW-UP

The question of why Cu forms face-centered closest packing, but Zn forms hexagonal closest packing is not answerable at this level. There seem to be long range forces with which one layer can influence the placement of a third layer.

Some common metals exhibiting face-centered closest packing are Al, Cu, Ag, Au, Pt, Ni, Ir, and Fe. Some exhibiting hexagonal closest packing are Ti, Be, Mg, Ca, Sr, Co, and Zn.

DEMONSTRATION 35

TWO WAYS OF STACKING LAYERS OF SPHERES

PURPOSE

To show the different types (lengths) of holes produced when spheres are packed in face-centered cubic or hexagonal closest packed arrangement.

TIMING

About 15 minutes are needed after Experiment 38.

MATERIALS NEEDED

- 22 spheres, 3 inch diameter, painted blue and glued to a white plywood base in a closest packed arrangement. One sphere must be removable. See Figure 32B.
- 15 spheres, 3 inch diameter, painted white and glued together with chenille stems to form a flat, closest packed layer.
- 15 spheres, 3 inch diameter, painted red and glued together with chenille stems to form a flat, closest packed layer.
- 1 sphere, 3 inch diameter, painted red and containing a $\frac{1}{4}$ inch hole along a diameter.
- 1 dowel, $\frac{3}{16}$ inch diameter and 10 inches long, with a handle glued to one end. See Figure 31.
- 5 spheres, 3 inch diameter, painted aluminum. This is the same as the 5-sphere unit described in Demonstration 34, Figure 29.

LAB HINTS

1. See Lab Hint 1 of Demonstration 34 for rolling and painting plus details on how to make the closest packed layers.

CLASSROOM PRESENTATION

Action	Comment
Show the blue, bottom layer.	Is this a closest packed layer? (Yes, each sphere has 6 nearest neighbors.) This is what is shown in Figure 17-17 in the Textbook.

Place the white layer on the blue layer.

Show the dowel with handle, and compare the dowel length to the height of two layers.

Call attention to the rows of holes. Place the single red ball on the white layer and over a hole that has no blue ball below it.

Put dowel through hole in single red ball.

Place the red layer on the white layer so that its spheres are in the same kind of holes the single red ball occupied.

Place single red ball on red layer, and show the dowel will drop down to touch a blue sphere in the bottom layer. Show there are no holes that go down to the base.

Remove the red layer and place the single red sphere on the white layer and over a hole which *has* a blue sphere under it.

Insert the dowel and show that it does not go down to the base.

Replace the single red sphere with the red layer so that its spheres are in the same kind of holes the single red ball just occupied.

Use the dowel (without sphere) to show there are some holes that go through the 3 layers, down to the base.

Here is a second closest packed layer. Compare this with Figure 17-18 in the Textbook.

The dowel is long enough to contact the base.

Notice the dowel will contact the base. This ball in the third layer is over a hole in the first layer. See Figure 31A.

This arrangement is called face-centered cubic and is often labeled 123123... because spheres in the three layers have their centers over different spots on the base.

Spheres in the fourth layer (the second "1" layer) are directly over spheres in the first layer.

This red sphere is over a blue sphere. See Figure 31B.

This arrangement is called hexagonal closest packed, or 121212... Each sphere in the red layer is directly over one in the blue layer.

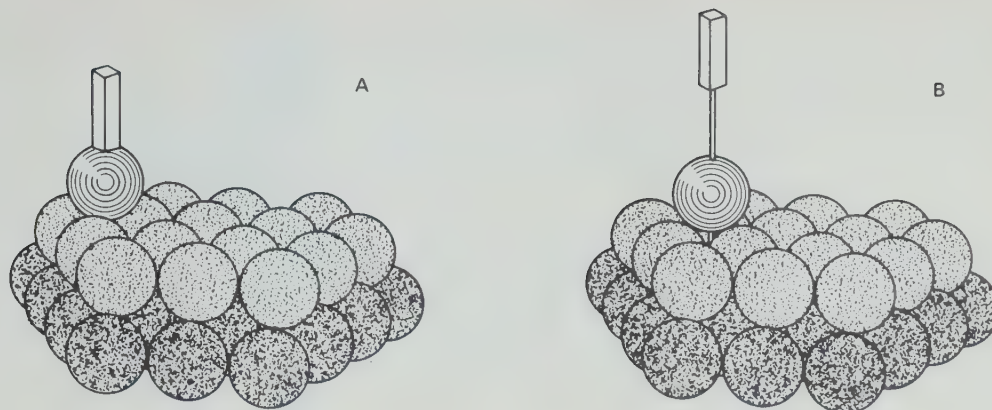


Figure 31
Two ways to stack close-packed layers.

You can also use this demonstration apparatus to show how a side of a face-centered cubic (f.c.c.) unit cell occurs in the layers of the 123123 . . . structure. For this you need a segment of the cell, as shown in Figure 32A.

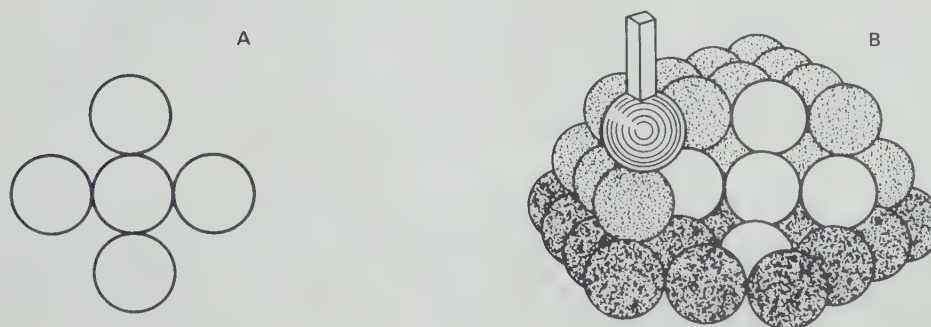


Figure 32
A. The special portion for showing the face-centered arrangement.
B. Face-centered cubic packing.

One ball of the first (blue) layer must be removable. The side of a f.c.c. unit cell will fit where the blue ball is removed if it is tilted to the proper angle. This is not possible if the layers are ordered 1212. . . . With the model in front of you, the arrangement is clear. Figure 32B shows the final view.

The Chemistry of Carbon Compounds



A high school text can only touch a few of the most important concepts of organic chemistry. Likewise, the laboratory program must be very elementary. Experiment 39 is designed to illustrate the major classes of hydrocarbons and to show some reactions of one functional group (—OH). Experiment 40 gives the student the chance to synthesize a compound and gain some experience with another functional group (ester).

Schedule of Experimental Work

TEXT SECTION	EXPERIMENTAL WORK	FOR DETAILS SEE PAGE
18-1 to 18-4	Expt. 39	227
	Expt. 40	232
18-5 to 18-14		

Experiment 39

Some Reactions of Hydrocarbons and of Alcohols

PURPOSE AND SUMMARY

To gain familiarity with some organic molecules. To provide illustrations of saturated, unsaturated, and ring compounds and of one functional group, hydroxide.

The student reacts cyclohexane, cyclohexene, benzene, and toluene with alkaline KMnO_4 , and with Br_2 . Ethanol is tested with neutral, acidic, and basic KMnO_4 . The oxidation of methanol is investigated. Three isomeric butanols are tested with Na, concentrated HCl, and neutral KMnO_4 .

TIMING

A full period is required anytime during Chapter 18. It is well to break this experiment into several parts. The number of chemicals confronting the student at one time is reduced, and the write-up is made easier. One day, do Part I—20 minutes (Questions 1 and 2 apply). Another day do Part II—35 minutes (Questions 3–7 apply).

MATERIALS NEEDED

per student

test tubes, 13×100 mm, minimum of 6, 9 desirable

test tube rack

asbestos square for smothering methanol fire

burner, ring stand, ring, wire gauze

distilled water

glass stirring rod

plastic thumb protectors

wax pencil or labels

The following should be available in dropper bottles

benzene, 1 ml

bromine in TTE, 5 ml 0.1 M (5 ml $\text{Br}_2(l)$ /995 ml TTE). See Lab Hint 1.

butanol, 4 ml each of primary, secondary, and tertiary

copper wire, 20 cm B and S #22

cyclohexane, 1 ml. See Lab Hint 2.

cyclohexene, 1 ml

ethanol, 1 ml

HCl, 15 ml 12 M , (conc. reagent)

H_2SO_4 , 2 ml 6 M , (340 ml conc. reagent/liter)

KMnO_4 , 14 ml 0.01 M , neutral (1.6 g KMnO_4 /liter)

methanol, 2 ml

NaOH, 4 ml 6 M , (24 g/100 ml).

sodium metal, 0.02 ml (about 0.02 g). See Lab Hint 3.

LAB HINTS

1. A solution of 5 ml Br_2 in 995 ml trichlorotrifluoroethane (TTE) is satisfactory (approx. 1% by mass), or dissolve in 150 ml TTE the bromine produced from 0.03 mole of NaBr (molar mass 103) or KBr (molar mass 119), oxidized by MnO_2 and H_2SO_4 . This amount is sufficient for a class of 30. Each student needs to use Br_2 in TTE quite a bit. Either have many bottles available, or provide a small container (crucible) and medicine dropper. The student can then take about 5 ml for his own use.
2. Do not leave these hydrocarbons in plastic squeeze bottles. When through with this experiment, store the chemicals in well-sealed bottles.

Other hydrocarbons may be substituted or added. Note that many chemicals have impurities and that it is especially difficult to avoid contamination by unsaturated hydrocarbons. With the quantities suggested, however, there will be a significant difference between the behavior of unsaturated compounds and that of liquids containing some unsaturated molecules as impurities. Test the chemicals you use. Avoid extremely volatile substances (about 75°C is the minimum safe boiling temperature).

3. Trim the oxide deposits from a small piece of Na that has been dried by blotting with a paper towel. Give each student three tiny slivers in separate, clean, dry test tubes. There will likely be some Na unreacted. To avoid the possibility of fire, have all excess solutions dumped into a clean waste container half full of ethanol.

PRELAB DISCUSSION

Have ball-and-stick models available so that students may study them. Point out that the four hydrocarbons are selected as representatives of classes of compounds. The reactions studied in this experiment help define these classes (saturated, unsaturated, and aromatic). Mention also that the reactions of the alcohols are characteristic and may be used to identify the functional group $-\text{OH}$.

Remind students about smothering methanol fires with an asbestos square (Part IId), and that water and sodium are not to be mixed.

A few students will feel that if colored Br_2 is added to colorless cyclohexene, and no color appears, then there is no evidence for a chemical reaction having taken place. Perhaps these few need special attention.

PRECAUTIONS

Place hydrocarbons and alcohols in small containers, and remind students that all are flammable. You should supervise the use and disposal of sodium. See Lab Hint 3. Have students wear safety glasses and aprons.

In Part II(e ii), special care is needed in shaking the mixture of alcohol and concentrated HCl. Relieve the pressure cautiously, while holding the tube away from the eyes.

POSTLAB DISCUSSION

Emphasize the relation between differences in reaction and differences in structure. Mention particularly the ease with which the double bond is oxidized and the differences in reactivity for various positions of the $-\text{OH}$ group.

SAMPLE DATA

PART I

The results of mixing alkaline KMnO_4 with some hydrocarbons are shown below.

		Cyclohexane	Cyclohexene	Benzene	Toluene
a)	After addition and mixing	purple (no change)	green (MnO_4^{2-})	purple (no change)	purple (no change)
	After 5 minutes	gray-purple (possibly some MnO_4^{2-})	brown ppt (MnO_2)	gray-purple (possibly some MnO_4^{2-})	gray (some MnO_4^{2-})
b)	<i>bromine</i> (see Note) After agitating	no change	becomes colorless ; continues to decolorize additional Br_2	no change	some slight loss of color

Note: Cyclohexene will remove the color of more bromine than will the other compounds. Their loss of or decrease in color is probably due to some unsaturated impurity in the compounds being furnished. But bromination of saturated compounds may be brought about when energy from sunlight is absorbed.

PART II

c) Ethanol with :	Basic KMnO_4	Neutral KMnO_4	Acid KMnO_4
after 10 seconds	green (MnO_4^{2-})	no change	red rather than purple
after 5 minutes	yellow-brown	no change or somewhat more red than purple	very light red or pink

d) The copper (wire or penny) remains hot and is alternately red-hot or black (oxide with possibly some carbon coating). The alternating reaction described here indicates oxidation-reduction ; the Cu may serve as a catalyst. Carbon dioxide and H_2O are produced plus some formic acid as well as formaldehyde. It is also interesting to try silver here ; it works quite well.

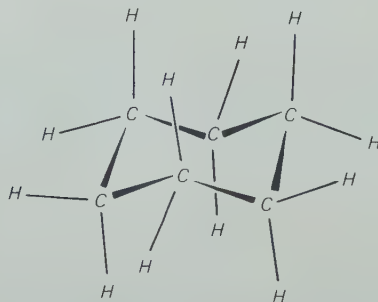
e) The results of mixing alcohols with various reagents are shown in the following table.

Reagent	Primary alcohol 1-butanol	Secondary alcohol 2-butanol	Tertiary alcohol 2-methyl-2-propanol
Sodium	bubbles of gas evident	fewer bubbles of gas than primary alcohol. Later, some white solid forms (sodium butoxide)	fewer bubbles of gas than primary alcohol. Later, considerable white solid forms (sodium <i>tert</i> butoxide)
Concentrated HCl	no reaction evident	no reaction evident	milky emulsion produced
Neutral KMnO_4 after first shaking	decided color change (deep red)	some color change (red)	some color change (to red)
After 5 minutes	brown ppt separates; liquid forms two layers, both clear	liquid in two layers; upper one is pink, lower one is brown-red with some ppt	single layer. A slight amount of brown ppt settles

RESULTS OF PROCESSING THE DATA

- 1) Examine the models of the various hydrocarbons you tested. Which contain double bonds? Which of the models are planar, which nonplanar? Is there an alternate structure for cyclohexane?

All but cyclohexane contain double bonds. Benzene and toluene are planar. Cyclohexane may be either "chair" (shown) or "boat" form.

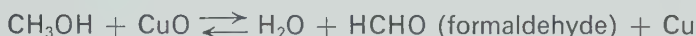


- 2) (a) Which of the hydrocarbons were readily oxidized by the alkaline solution of KMnO_4 ?
 (b) Which was reactive with the bromine solution?
 (c) What is the relation between the reactivity noted in (a) and (b) and the structure of the hydrocarbons?
- (a) Cyclohexene was most readily oxidized.
 (b) Cyclohexene was most reactive.

This is the only one that is expected to react, but some unsaturated compounds are nearly always present as impurity in the supposedly saturated samples. The benzene ring compounds do react with bromine, not as addition of bromine, but as a substitution for a hydrogen atom. This, however, does not take place under ordinary

conditions unless a catalyst is present. There is no reason why reaction would be more likely with toluene than with benzene, hence if a reaction is observed, it probably reflects the lack of purity in the substances used.

- (c) These reactions show that an isolated double bond (as in cyclohexene) is not the same as a double bond in an aromatic ring (benzene). Many other results reinforce this conclusion. The bonding is different, as hinted at in the Textbook discussion (p. 363) about the symbol for benzene.
- 3) Write a balanced equation for the reaction in which methanol is oxidized by the hot copper oxide.

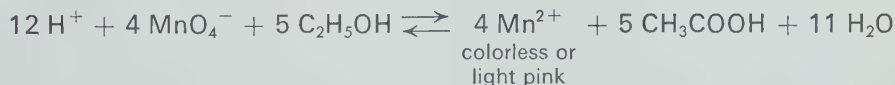


That there was no explosion argues against H_2 and O_2 being products.

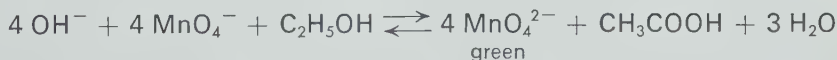
- 4) What differences were noted when $\text{C}_2\text{H}_5\text{OH}$ reduced KMnO_4 in neutral, acidic, and basic solutions? Assuming that $\text{C}_2\text{H}_5\text{OH}$ was oxidized to acetic acid in each case, write balanced equations for each reaction. Be sure to use the reduction half-reaction that involves the reduction product of manganese which you observed.

The general comment that manganese has several oxidation states accounts for the differences noted. The rates of these reactions are strongly affected by H^+ and are very slow in neutral solutions.

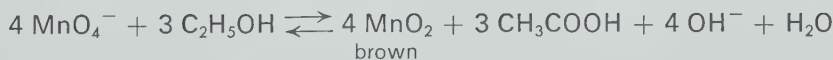
In strong acid solution :



In basic solution, after a few seconds :



after 5 minutes :



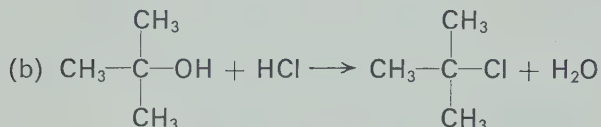
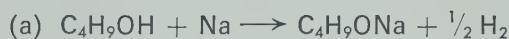
In neutral solution, the student will have observed very little reaction, hence should probably not write an equation. It may be, however, that the distilled water is slightly acid and that some Mn^{2+} is produced. If alkaline tap water is used, some brown MnO_2 may have resulted.

- 5) In the reactions involving the three isomeric alcohols with the formula $\text{C}_4\text{H}_9\text{OH}$, what did each of the following tests show about the activity of the functional group $-\text{OH}$ and its position in each alcohol?
- (a) The test with metallic sodium ;
- (b) The test with concentrated hydrochloric acid ;
- (c) The test with neutral potassium permanganate.



All the alcohols showed the same reaction, but the primary alcohol was more reactive, and the product, RONa , from the tertiary was least soluble. The RONa compounds are called sodium alkoxides, in this case butoxides.

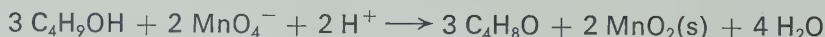
- (b) For 2-methyl-2-propanol a reaction occurs, producing a product of low solubility. The others showed no reaction. This indicates a less strong bond between the C and the OH in the compound where the carbon is bonded to three other carbons.
- (c) The primary and secondary alcohols were more easily oxidized. Aldehydes and acids were produced from the primary alcohol and a ketone from the secondary alcohol (shown also in the film "Synthesis of an Organic Compound").
- 6) Write a balanced equation for each case in Question 5(a), (b), and (c) where a reaction occurred.



- (c) Using the oxidation number system we see that the carbon to which oxygen is attached goes from +2 to 0, while manganese goes from +7 to +4. Hence three molecules of alcohol must react for each two MnO_4^- ions:

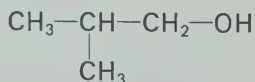


Using H^+ and H_2O to balance gives



- 7) *Optional:* There is a fourth alcohol with the formula $\text{C}_4\text{H}_9\text{OH}$. Draw a structural formula for it and name it. How would you predict that it would react with
- (a) metallic sodium;
- (b) 12 M HCl;
- (c) neutral 0.01 M KMnO_4 ?

The fourth isomer, 2-methyl-1-propanol, is



It would be expected to react as the primary alcohol did—with Na and with KMnO_4 , but not with HCl.

Experiment 40

The Preparation of Esters

PURPOSE AND SUMMARY

To prepare two esters and become familiar with two new laboratory techniques: refluxing and distillation.

The student makes ethyl acetate and methyl salicylate.

TIMING

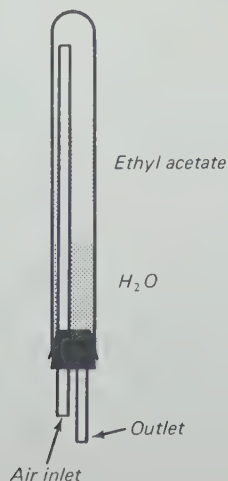
About 50 minutes are needed ; before Section 18-5.

MATERIALS NEEDED**per student**

- 1 beaker, 250 ml
- boiling chips
- 1 hot plate and/or ring stand, clamps, burner
- 1 separatory funnel. See Lab Hint 1.
- 1 small condenser. See Lab Hint 2.
- 1 test tube, 25 × 200 mm, with a 1-hole stopper and a 2-foot length of 8–10 mm glass tubing. See Lab Hint 3.
- 6 test tubes, 13 × 100 mm
- acetic acid, glacial, 6 ml
- calcium chloride, anhydrous, 0.5 g. See Lab Hint 4.
- ethanol, 5 ml
- H₂SO₄, 0.5 ml 18 M (for safety in dispensing see p. 122, Lab Hint 3, Expt. 24)
- methanol, 10 ml
- salicylic acid, 3 g
- sodium carbonate, saturated, 2 ml (about 20 g Na₂CO₃·H₂O/100 g water)

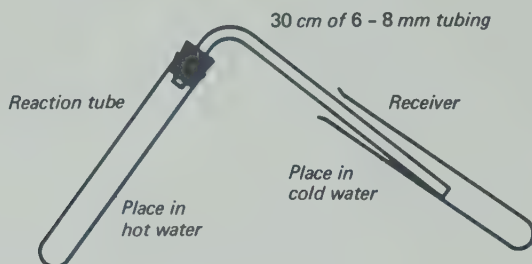
LAB HINTS

1. If small separatory funnels are not available and you wish to introduce separation of liquids, a substitute may be made as follows :



Remove the stopper, and place the liquid in the tube. Invert the tube, keeping a finger over the outlet (or provide other means of closure). When the liquids have formed layers, control the outflow of heavier liquid by finger pressure on the air inlet tube.

2. A small water-cooled distilling condenser is desirable for Part I, but if such is not available, a right-angle bend may be used as follows:



3. A cork is better than a rubber stopper because of the greater ease of boring a suitable hole for 8–10 mm tubing. If rubber is used, see Lab Hint 5. If cork is used, wrap the lower part in aluminum foil so that vapors will not be absorbed.
4. In place of anhydrous calcium chloride, either anhydrous magnesium sulfate or Drierite may be used.
5. Here is a safe method for putting a thermometer or glass tube through a rubber stopper. Select a cork borer of a size that the thermometer will just slide through. After moistening the hole in the stopper with a drop or two of glycerol, work the cork borer into the stopper hole from the bottom. When it is through, slide the thermometer in from the top until it extends the desired distance. Hold the stopper and thermometer, and work the cork borer out. At first the thermometer will need to be held in position, but as soon as the cork borer is a little way out, the rubber will grip it. The thermometer may be removed in a similar fashion by working the borer down around it. This works even for thermometers welded to the rubber.

PRELAB DISCUSSION

Have ball-and-stick or other molecular models available. Emphasize the integrity of the hydrocarbon group during changes of functional group. The reactions are characteristic of the functional groups rather than of the entire molecule. Caution students not to paralyze their olfactory nerves by inhaling too much chemical.

PRECAUTIONS

Ethanol, ethyl acetate, and methanol are flammable; therefore heating must be done very carefully. Electric hot plates are preferred. Be sure to caution students about the use of concentrated H_2SO_4 . See p. 123 for convenient dispenser. Safety glasses and aprons should be worn.

POSTLAB DISCUSSION

This is an appropriate time to discuss ways of purifying organic compounds—distillation, crystallization, use of differential solvents. It is also good to comment on the use of equilibrium considerations in organic reactions.

A few esters and the odors of which they are characteristic, are listed below.

amyl or isoamyl acetate—bananas

amyl butyrate—apricots

amyl formate—plums

butyl butyrate—pineapples

SAMPLE DATA

PART I

- c) Ethyl acetate, if dilute enough, has the odor of apples.
- d) With the water bath kept at 90°C, about 7–8 ml evaporate and are condensed. It is assumed that what is left behind is the unreacted acetic acid, water, and sulfuric acid. Along with the ethyl acetate will be some unreacted ethanol, since this boils at 79°C and the ethyl acetate at 77°C. The boiling temperature of the acetic acid is 118°C.
- e) The ethyl acetate has a solubility of 8.6 g/100 g water at 20°C, but with saturated sodium carbonate the separation is fairly good. Less than two milliliters of Na₂CO₃ solution will be needed if the temperature has been kept as suggested in Part d. The water layer will be below the ethyl acetate, since the density of the latter is 0.9 g/ml.
- f) It is unlikely that the student will have more than 5 or 6 milliliters at this point (5 ml of product, if all ethyl acetate, would be 0.05 mole or about 60% yield), and there is not much need to dry or further distill it. The most noticeable property is the odor. A sample of commercial ethyl acetate should be put out for comparison. This compound is a good insecticide and is, in fact, used instead of cyanide in insect-killing jars for entomological collecting. The student could investigate its effectiveness by comparing it with ethanol.

RESULTS OF PROCESSING THE DATA

- 1) (a) Calculate the number of moles of each reactant, C₂H₅OH and CH₃COOH, which was used in the preparation of CH₃COOC₂H₅.

Data: 1 ml of C₂H₅OH weighs 0.79 g ;

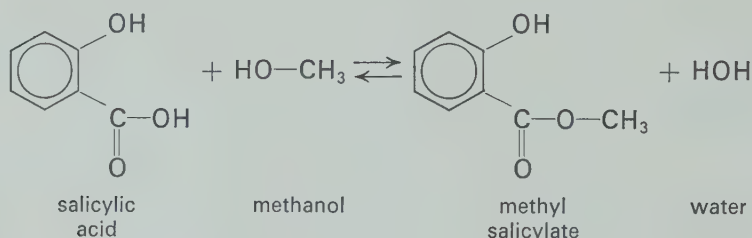
1 ml of CH₃COOH weighs 1.05 g.

- (b) Which reactant is present in excess ?
 - (c) If all of one of the reactants were consumed, how many moles of ethyl acetate could be produced ? How many grams of CH₃COOC₂H₅ is this ?
 - (d) What is the role of the sulfuric acid in the reaction ?
- (a) $\text{CH}_3\text{COOH} + \text{C}_2\text{H}_5\text{OH} \rightleftharpoons \text{CH}_3\text{COOC}_2\text{H}_5 + \text{H}_2\text{O}$

	Acetic Acid	Ethanol
Mass of 1 mole	60 g	46 g
Density	1.05 g/ml	0.79 g/ml
Mass used	6.30 g (6 ml)	3.95 g (5 ml)
Moles	0.105	0.0859

- (b) The acetic acid is therefore in excess, since ethanol and acetic acid react 1 mole to 1 mole.
- (c) If all the ethanol (the reactant not in excess) were used, 0.0859 mole of ethyl acetate could be produced. This would be 0.0859×88.0 g/mole, or 7.56 grams. Since its density is 0.9 g/ml, this will be 8.4 ml.
- (d) The sulfuric acid reacts with the water produced, and hence acts as a dehydrating agent. This is an example of Le Chatelier's Principle. By removing a product, the equilibrium state is caused to favor products to a greater extent.

2) Write the equation representing the reaction between salicylic acid and methanol to form the ester



3) *Optional.* Explain the observations made in Step e concerning the different odors from the different tubes.

Each tube will contain a mixture of reactants and products. The chemical with lowest boiling temperature will boil off first.

	boiling temperature, °C
ethyl acetate	77.2
ethanol	78.4
water	100.0
acetic acid	118.1
sulfuric acid	decomposes at 340

The ethyl acetate odor should be strongest in the first tubes.

The Halogens



Oxidation-reduction reactions are a most important class for the halogens. They are studied electrolytically (Experiment 41) and chemically (Experiment 42).

Schedule of Experimental Work

TEXT SECTION	EXPERIMENTAL WORK	FOR DETAILS SEE PAGE
19-1 to 19-4	Expt. 41	238
	Expt. 42	240
19-5, 19-6		

Experiment 41

The Electrolysis of Aqueous Potassium Iodide

PURPOSE AND SUMMARY

To identify the products of electrolysis of a 0.5 *M* potassium iodide solution and to study some of the chemistry of iodine.

The student prepares an electrolytic cell using 0.5 *M* potassium iodide solution. After electrolysis, he identifies the products of the reaction, using selected reagents and indicators. From their observations and tests the students are able to write equations for the reactions.

TIMING

Assign this experiment anytime after Section 19-4. A full 45–50 minutes are required to complete the experiment. There are convenient stopping places at any point after step a, providing the apparatus can be left set up.

MATERIALS NEEDED

per group of students

- 1 U-tube. See Lab Hint 1.
- 1 D.C. source, 12 volts. See Lab Hint 2.
- 2 electrodes, carbon rod
- 2 wires, insulated, bell wire with end clips
- 1 ring stand and clamp
- 1 pipet or medicine dropper
- plastic thumb protectors
- ferric chloride solution, 3 ml 0.1 *M*, in dropper bottle
(2.7 grams $\text{FeCl}_3 \cdot 6 \text{H}_2\text{O}$ /100 ml)
- phenolphthalein solution, few drops, 1.0%
(1.0 gram/100 ml 50% ethanol)
- potassium iodide solution, 100 ml 0.5 *M*. See Lab Hint 1.
(83 grams KI/liter of solution)
- 2 ml trichlorotrifluoroethane (TTE)

LAB HINTS

1. A large range of U-tube sizes will do. The amount of 0.5 *M* potassium iodide needed depends on the volume of the U-tube used.
2. Your physics department may be able to supply D.C. battery eliminators. See Lab Hint 3, Experiment 36, p. 204 in this guide.

PRELAB DISCUSSION

None necessary.

PRECAUTIONS

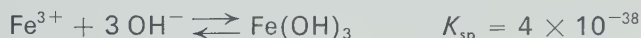
Take precautions appropriate to your D.C. source. Iodine may stain fingers, but may be removed if sodium thiosulfate is used promptly. Use a solution, or moisten a few crystals held in the palm of the hand, and rub the stained finger in this.

SAMPLE DATA

The observations as such are very interesting, but the student will have difficulty interpreting what happens. He will observe the brown color streaming down from the anode and filling the base of the U-tube. For Part b he will surely write $2 \text{I}^- \rightleftharpoons \text{I}_2 + 2 \text{e}^-$ and, if encouraged to recall Experiment 34, Part II, he will realize that I_2 is not very soluble in water. In Question 2 the student is told that $\text{I}_2 + \text{I}^- \rightleftharpoons \text{I}_3^-$.

After using the indicator he should have no difficulty identifying the reaction at the cathode as $2 \text{H}_2\text{O} + 2 \text{e}^- \rightleftharpoons \text{H}_2 + 2 \text{OH}^-$, and he will be able to decide this on the basis of E° values.

For Part c the equation for the formation of the precipitate is



The distinct odor of iodine is observed coming from the anode when it is removed. TTE turns purple when the anode is placed in it. Additional ferric chloride reacts with I^- according to the equation



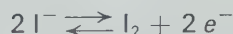
The FeCl_3 should be added slowly. $\text{Fe}(\text{OH})_3$ is first produced, then I_2 . Added TTE turns purple.

For Part d he will observe the lavender, and finally deep purple, color of the iodine in TTE. Experiment 34 gave the student experience with this test.

A number of extensions of this experiment may be of interest if time permits. A one milliliter sample of solution at the cathode may be removed at ten-minute intervals and titrated (0.1 M HCl is suitable). The electrodes may be reversed, the tube may be clamped at different angles, the depth of the electrodes may be changed, the concentration of the KI solution may be varied, and the solution may be stirred.

RESULTS OF PROCESSING THE DATA

- 1) Write the equation for the anode half-reaction.



- 2) As iodine is produced at the anode, it reacts with iodide ions to form the complex triiodide ions, I_3^- . These complex ions cause the electrolyte solution to turn brown. Write the equation for this reaction using double arrows to represent equilibrium. What effect did the addition of TTE have on the equilibrium? Use your observations of the changes in color of the two layers as clues to explain the effect.



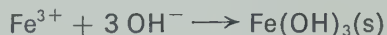
Since I_2 is very soluble in TTE, the equilibrium is shifted to the left, and the deep purple color is obtained in the lower layer while the color in the aqueous layer fades, since the I^- left there is colorless.

- 3) Is the solution around the cathode acidic or basic? Write the equation for the cathode half-reaction. Write the equation for the reaction of ferric chloride solution with the sample of solution taken from the cathode side of the U-tube.

Phenolphthalein turns pink indicating the solution is basic. The cathode reaction is represented by the equation



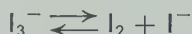
The equation for the reaction with ferric chloride is



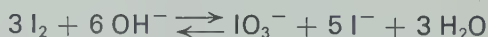
- 4) When iodine reacts with a basic solution, it undergoes a self-oxidation-reduction reaction to form iodide ions and iodate ions—both of which are colorless in aqueous solution. Give an explanation of the sharp color boundary noted near the bottom of the U-tube in terms of your knowledge of the products at each electrode. Write the equation for the reaction involved.

If the student has observed carefully, he will note that gravity strongly influences the accumulation of the I_2 and thereby the I_3^- in the bottom of the U-tube. Stirring is limited in this region. Further observation on this may be obtained by tipping the U-tube, and possibly by raising and lowering the electrodes, or by stirring the solution.

The boundary is kept "sharp" by a balance between the diffusion rates of OH^- (downward) and I_3^- (upward), plus the reactions



and



Experiment 42

Some Chemistry of Iodine

PURPOSE AND SUMMARY

To study oxidation-reduction reactions involving compounds of a typical halogen. To study the effect of changing the hydrogen ion concentration on some equilibrium reactions of iodine. To be able to deduce the products of reactions as identified by known tests and write equations representing the observed reactions.

The student combines solutions of iodine compounds with various oxidizing and reducing reagents. In Part I, solutions of potassium iodide are reacted with various reagents. In Part II, solutions of potassium iodate are used. Part III gives the student experience with reactions of I_2 in basic solution.

TIMING

The experiment can be assigned anytime after Section 19-4 in the Textbook.

At least one full laboratory period will be required.

MATERIALS NEEDED**per student or pair**

3 or more test tubes, 13 × 100 mm

1 test tube, 18 × 150 mm, Pyrex.

See Lab Hint 1.

1 stirring rod

1 test tube holder

1 funnel and filter paper, or centrifuge

1 burner, ring stand, and clamp

iodine crystals, 1 gram

potassium iodate, 0.5 gram, (KIO_3)

sodium metabisulfite, 1.2 gram, ($\text{Na}_2\text{S}_2\text{O}_5$)

per class

The following should be supplied in dropper bottles. See Lab Hint 2.

KOH, 3–4 ml 6 *M* (33.6 g/100 ml)

HNO_3 , 1 ml 6 *M* (39.4 ml conc./100 ml)

AgNO_3 , 3 ml 0.1 *M* (1.7 g/100 ml)

KIO_3 , 10 ml saturated (about 10 g/100 ml)

H_2SO_4 , 2 ml 6 *M* (34 ml conc./100 ml)

KI, 12 ml 0.1 *M* (8.3 g/500 ml)

starch solution, 15 ml (2 g/500 ml).

See Lab Hint 3.

NaOCl , 0.5 ml 5% (commercial bleach)

H_2O_2 , 0.5 ml 3%

LAB HINTS

1. Note that for Part II e, an 18 × 150 mm tube is called for so there will be greater condensing surface for the iodine vapors and more reactant surface from which vapors may escape.
2. The solutions should be used in dropper bottles. KOH should not be stored in bottles with ground glass stoppers. HNO_3 should not be stored for a long time in a dropper bottle with a rubber bulb.
3. Prepare soluble starch solution by adding 2 grams soluble starch to 500 ml boiling water.

PRELAB DISCUSSION

Preliminary Experiment. Discuss the starch– I_2 test with the students. This test will reveal the presence of 1×10^{-7} gram of iodine per milliliter. When the water containing excess iodine crystals is warmed, students will observe some purple vapors rise above the water. Usually a faint color develops in the water, which may be due to the slight dissolution of I_2 . It is more likely, however, that the chlorination of the water gives enough Cl^- to form a complex with the iodine and that this is responsible for the dissolution of I_2 and the resultant color. Tap water develops this color, but properly distilled water does not.

PRECAUTIONS

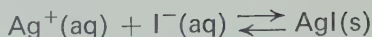
Solid and gaseous iodine will stain skin and clothing. These vapors irritate mucous membranes. Be sure to caution students to heat the tube in Part II e very gently. Good ventilation should be provided. Use a fume hood if available. Use safety glasses and aprons.

POSTLAB DISCUSSION

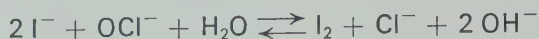
Discuss student observations.

PART I

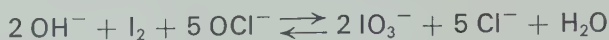
- a. A heavy cream-yellow precipitate forms.



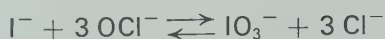
- b. When a drop or two of bleach is added, a blue color appears, showing that the bleach has oxidized the I^- to I_2 .



When additional bleach is added, the color disappears, since the I_2 is further oxidized to IO_3^- .

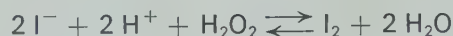


Or, in one step, the reaction is



If desired, the student could test for the Cl^- by adding AgNO_3 to the bleach alone and to a comparable amount of the product of this reaction. Since the action of the NaOCl with water is such that OH^- is produced, one may also interpret the disappearance of the I_2 as being due to the OH^- (see discussion of Part III). If acid is added with the bleach, the color does not disappear.

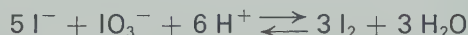
- c. With H_2O_2 , I_2 forms as revealed by the starch indicator. The reaction is



The appearance of color is delayed and depends upon $[\text{H}^+]$. With base added, no evident reaction takes place, and with the addition of acid, it is much increased. It may be that, with base, IO_3^- is formed.

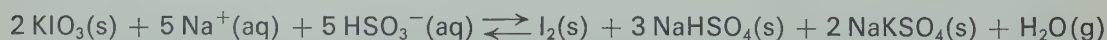
PART II

- d. A thick, dark-brown precipitate forms. After filtering or centrifuging this from the liquid, the solid will be identified as iodine. To test, add a little water and starch solution. Part II of Experiment 34 may be recalled and trichlorotrifluoroethane (TTE) used to give the characteristic violet solution.

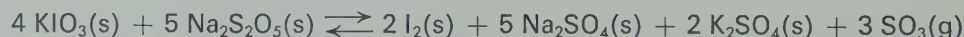


Since basic solution (that is, OH^-) favors the formation of I^- and IO_3^- in this reaction, there is no change here. The conclusion is that H^+ is necessary for I^- and IO_3^- to produce iodine.

- e. When solid KIO_3 is heated with $\text{Na}_2\text{S}_2\text{O}_5$, a brown color is observed, soon followed by fumes of gaseous I_2 . This will condense to the characteristic, shiny, gray solid on the cooler portions of the test tube. The student cannot be expected to write the equation for this:



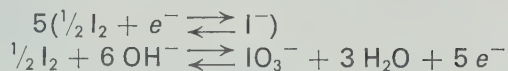
Although the student actually added $\text{Na}_2\text{S}_2\text{O}_5$, water reacts with this compound to produce $\text{Na}^+(\text{aq}) + \text{HSO}_3^-(\text{aq})$. Another form of the equation is



There will almost certainly be some moisture in the air, with the result that H_2SO_4 rather than SO_3 will be the final product.

PART III

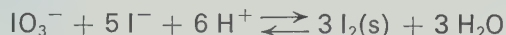
- f. The iodine dissolves, giving a colorless solution :



The net reaction is



- g. When acid is added, a deep brown-black solid results. This is $\text{I}_2(\text{s})$.



- h. The solid iodine redissolves in KOH as in f.

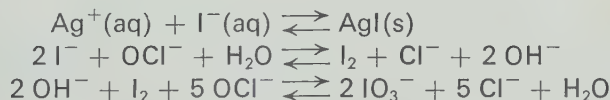
- i. When the solution containing I_2 in KOH is cooled, a small amount of white solid (KIO_3) is formed.

- (1) When this is dried and heated with $\text{Na}_2\text{S}_2\text{O}_5$, the brown color characteristic of I_2 will be noted, and some I_2 vapor (lavender) will be produced (see Part IIe).
- (2) Yellow AgI forms as a cream-yellow precipitate when AgNO_3 is added to the decanted liquid. This will be enough like Part I a to be recognized, although the presence of OH^- will result in *some* brown $\text{Ag}_2\text{O}(\text{s})$.

Point out that the other halogens display similar reactions. Show some of these—for example, the reactions of each halogen with Br^- , NaOCl , and H_2O_2 , and reactions for the bromate and chlorate ions.

RESULTS OF PROCESSING THE DATA

- 1) Write the equations for the reactions observed in steps a, b, and c.



- 2) How did the results in step i(i) compare with those obtained in step e? How did the test with silver nitrate solution in step i(ii) compare with the results of step a? What do you conclude about the ionic species formed when iodine reacts with potassium hydroxide solution as in step f?

They were very similar. Both had the brown color characteristic of $\text{I}_2(\text{s})$ and the lavender vapor of $\text{I}_2(\text{g})$.

A cream-yellow precipitate formed [see discussion for Part III i(ii)].

Both I^- and IO_3^- are formed.

- 3) Write the equation for the self-oxidation-reduction reaction of iodine in a basic solution. Write the equation for the reverse of this reaction in acidic solution.



The Fourth-Row Transition Elements



Transition elements provide some of the most distinctive compounds and intricate chemistry that occurs for noncarbon substances. These elements include some of the commonest and rarest ones known. In the four experiments provided the student learns some new techniques (ion exchange, Experiment 43; crystallization, Experiment 44) and prepares inorganic compounds (Experiments 44–46).

Schedule of Experimental Work

TEXT SECTION	EXPERIMENTAL WORK	FOR DETAILS SEE PAGE
20-1 to 20-3		
20-4	Expt. 43	245
	Expt. 44	249
20-5		
	Expt. 45	252
	Expt. 46	255

Experiment 43

The Separation of Iron, Cobalt, and Nickel Ions With an Anion Exchange Resin

PURPOSE AND SUMMARY

To illustrate a modern method for separation and analysis of ions in a solution and to give an opportunity for the discussion of some complex ions.

The student puts Fe^{3+} , Co^{2+} , and Ni^{2+} ions on a column and then removes them separately in the order Ni^{2+} , Co^{2+} , Fe^{3+} as the column is washed with progressively more dilute hydrochloric acid.

TIMING

A full period is needed during Section 20-4.

MATERIALS NEEDED

per student

50 ml 9 M HCl (770 ml conc. acid/liter)

ion exchange column. See Lab Hint 1. 10 cm column of resin in a 10–12 mm tube 20–30 cm long

stopper for column, rubber connector, pinch or screw clamp, and 2 short lengths of glass tubing, each 5 cm long. See figure in Lab Manual, p. 112.

2 ml test solution containing Fe^{3+} , Co^{2+} , and Ni^{2+} . See Lab Hint 2.

About 1 ml each of the following solutions

KSCN, 0.1 M (0.97 g/100 ml)

$\text{NH}_3(\text{aq})$, 15 M (conc. reagent)

One sample each of Fe^{3+} , Co^{2+} , and Ni^{2+} solution for color comparison. See Lab Hint 3.

1% dimethylglyoxime in ethanol (1 g/100 ml 95% ethanol)

10% NH_4SCN in acetone (10 g/100 ml)

ring stand, buret clamp

plastic film (approx. 10 × 10 cm)

3 small (50–100 ml) beakers or other containers for acid

12–15 test tubes, 13 × 100 mm

test tube rack

litmus paper

LAB HINTS

1. Preparation of the column:

Analytical grade (AG2-X8) resin is recommended. This is Dowex anion resin No. 2, which has been carefully washed and sized. A mesh of 50–100 is convenient for this experiment.

This resin is available from Bio-Rad Laboratories, 32nd and Griffin, Richmond, California, 94804. It is shipped slightly moist.

Caution: If dry resin is used, take care not to breathe any of the powder.

- (i) Use 10–12 mm tubing about 20 cm long. Ten grams of resin will occupy half the tube, leaving a reservoir space for solutions. Insert an exit tube into a small stopper, and clamp it as in Lab Manual Figure 43-1; have another stopper available to close the top when the column is left overnight. You can pull the glass tubing down to a small diameter and avoid the trouble of putting a tube through the small stopper. A buret can be used in place of the tubing and clamp assembly.
- (ii) Make a slurry of resin in 2 *M* HCl, and have extra acid available.
- (iii) Insert a glass wool plug just above the stopper (see Figure 43-1).
- (iv) Pour approximately the correct amount of slurry into a test tube and then into the column. The extra acid will flow out quickly.
- (v) Insert another glass wool plug.
- (vi) Close the exit tube, and add enough acid to cover the resin and the upper plug. The columns may be stored from one year to the next if kept moist. The best method is to wash the columns with water and place them (with clamp removed but top stopper in place) in a jar of water. A one-gallon wide-mouth jar (mayonnaise or pickle jars are available from school cafeterias) will hold a number of columns. One day before the next use, recondition the tubes by running 2 *M* HCl through them.

In such a tube the flow rate will probably need to be adjusted slightly by cutting down the opening with a pinch clamp. A rate of 2.5 ml per minute is adequate for washing, and allows enough time to record details. If by any chance the rate is too slow, aspirators may be used, or you may have the student attach a rubber squeeze bulb to apply pressure.

2. Preparation of test solution:

Under good conditions the analytical grade resin will hold 1.4 milliequivalents/ml [$(1.4 \times 10^{-3})(0.33)$ mole Fe^{3+} or $(1.4 \times 10^{-3})(0.5)$ mole Co^{2+} or Ni^{2+} , but there is no point in loading it completely for this experiment. The experiment has been designed for the use of 2 ml of solution containing 0.05 *M* Fe^{3+} , 0.1 *M* Co^{2+} , and 0.1 *M* Ni^{2+} .

Add 1.4 grams $\text{FeCl}_3 \cdot 6 \text{H}_2\text{O}$, 2.4 grams $\text{CoCl}_2 \cdot 6 \text{H}_2\text{O}$ and 2.4 grams $\text{NiCl}_2 \cdot 6 \text{H}_2\text{O}$ to 100 ml 9 *M* HCl.

or

Add 10 ml 1 *M* CoCl_2 and 10 ml 1 *M* NiCl_2 and 5 ml 1 *M* FeCl_3 to 75 ml of 12 *M* HCl (conc. reagent).

3. Since 2 ml of solution of each ion are diluted to about 10 ml in the process of elution, the 0.01 *M* Fe^{3+} , 0.02 *M* Co^{2+} , and 0.02 *M* Ni^{2+} solutions are reasonable for color comparison. The Co^{2+} should be shown also in 9 *M* and in 5 *M* (or less) HCl to show the contrasting blue and pink solutions when the degree of aqutation varies. Make them as follows.

0.01 <i>M</i> Fe^{3+}	dissolve 2.7 g $\text{FeCl}_3 \cdot 6 \text{H}_2\text{O}$ in 1 liter 1 <i>M</i> HCl (86 ml conc./liter)
0.02 <i>M</i> Co^{2+}	dissolve 4.8 g $\text{CoCl}_2 \cdot 6 \text{H}_2\text{O}$ in 1 liter 5 <i>M</i> HCl (430 ml conc./liter)
0.02 <i>M</i> Ni^{2+}	dissolve 4.8 g $\text{NiCl}_2 \cdot 6 \text{H}_2\text{O}$ in 1 liter 9 <i>M</i> HCl (770 ml conc./liter)

PRELAB DISCUSSION

The use of an ion exchange resin for separating and identifying the transuranium elements is similar to what is done in this experiment and may interest the students. Such resins are a useful tool in research. Make it clear that we only appear to be separating cations; actually an anion exchange is being used, since negative chloride complexes are involved. The sample is converted to a maximum concentration of these anionic complexes by addition of a high concentration of the anion that forms the complex (in this experiment, Cl^-). Then as the concentration is gradually decreased the complexes dissociate (the weakest one first) to give the hydrated metal cations which are no longer retained by the resin.

The colors of the hydrated ions being eluted will be too faint to be noticeable in the tube leading from the column, but will probably show up in the collecting tubes if viewed, as suggested, against a white paper background.

PRECAUTIONS

Observe the usual laboratory precautions, particularly with concentrated HCl and $\text{NH}_3(\text{aq})$. Have the students wear safety glasses and aprons.

POSTLAB DISCUSSION

A discussion of complex ions is appropriate as a follow-up of this experiment. Students may also be interested in knowing that chloride complexes of elements from Mn through Zn may be separated in a similar manner. See Kraus and Moore, *J. Am. Chem. Soc.*, **75**, 1460 (1953).

SAMPLE DATA

The third 5 ml portion of eluant in step (e) will almost certainly be colorless, but if not, any remaining Ni^{2+} will come off with the first of the 5 M portions. After adding four 5 ml portions of 5 M acid in (f), any Co^{2+} left (shown by a green color on the column) will wash off with the first of the 1 M rinsings.

If a trace of Fe^{3+} is left on the column after four or five portions of 1 M acid have been used, this will not interfere with further use of the column. You should make one or two test runs before assigning this experiment to the class, since samples of resin can differ considerably.

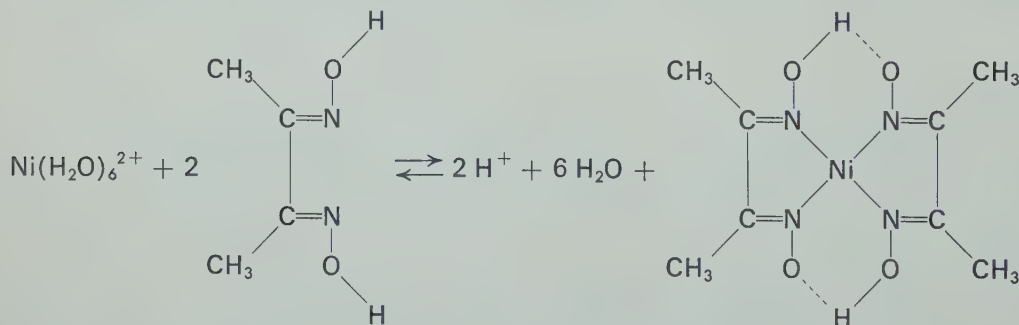
The following data were obtained with 10 cm of AG2-X8 resin in a 10 mm diameter tube. Five-ml samples were collected and tested as described in the Laboratory Manual. The flow rate was 2.5 ml/minute.

Eluate Tube No.	Acid	Color of Eluate	Result of Chemical Test for		
			Ni ²⁺	Co ²⁺	Fe ³⁺
e-1	9 M HCl	green	+	—	—
e-2		green	+	—	—
e-3		none	—	—	—
f-1	5 M HCl	none	—	—	—
f-2		blue	—	+	—
f-3		pink	—	+	—
f-4		pale pink	—	+	—
g-1	1 M HCl	none	—	sl+*	sl+*
g-2		yellow	—	—	+
g-3		yellow	—	—	+
g-4		pale yellow	—	—	+
g-5		none	—	—	sl+

* The KSCN gave a pale red color; the NH₄SCN, a gray color.

The qualitative tests for the various ions are as follows:

- (j-1) Fe³⁺(aq) with KSCN gives blood-red FeSCN²⁺ and probably some higher complexes [see *J. Chem. Education*, **30**, 445 (1953)].
- (j-2) Ni²⁺(aq) produces a brilliant red precipitate with dimethylglyoxime in alkaline solution.



- (j-3) Co²⁺(aq) with NH₄SCN gives a blue color, probably Co(SCN)₄²⁻. It is pink when hydrated to Co(SCN)₄(H₂O)₂. The function of the acetone is to lower the water concentration enough to stabilize the blue tetrahedral complex.

RESULTS OF PROCESSING THE DATA

- 1) One of the metallic ions was eluted with 9 M HCl. It would also have been eluted with the 5 M or 1 M HCl solutions. Which one was it? Give a reason for this ease of elution in terms of the stability of its complex chloride anions.

The Ni²⁺ does not form a complex chloride anion that is stable enough to be held on the column by the resin. The student may suggest the possible formation of NiCl₄²⁻, but this complex has not been specifically reported.

- 2) The color of some of the ions on the column was different from the color of the final eluate. Explain this in terms of the composition of the complex ions.

For the metallic ions to be held by the resin, a complex chloride ion must be formed. The complex may differ in color from the aquated ion. For cobalt, the chloride complex on the column seems to be green, although this may be due to the mixture of a blue complex with the yellow Fe^{3+} complex. In the concentrated acid the cobalt complex contains chloride and is blue in color. This complex is probably a mixture of CoCl_4^{2-} and $\text{CoCl}_3(\text{H}_2\text{O})^-$. [See Cotton and Goodgaine, *J. Am. Chem. Soc.*, **83**, 4690 (1961).] The species for the iron complex is FeCl_6^{3-} , which is yellow.

- 3) What are the conditions which favor the release of ions from the column?

The ions are released from the column when the concentration of chloride ion is too low for the complex to be stable.

- 4) Recalling that the resin used is an anion exchange type, which of the metallic ions in this experiment forms the most stable complex chloride anion?

The FeCl_6^{3-} is the most stable complex chloride used in this experiment.

Experiment 44

Preparation of A Complex Salt

PURPOSE AND SUMMARY

To give experience with the preparation of a complex salt and with crystallization. The student prepares $\text{Cu}(\text{NH}_3)_4\text{SO}_4 \cdot \text{H}_2\text{O}$ from $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and $\text{NH}_3(\text{aq})$. Crystals are grown and their behavior toward dissolving and being heated is tested by comparing with that of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$.

TIMING

The experiment fits well with Section 20-4. Parts 1 a–c require 20 minutes and need to be followed by overnight crystallization. Parts 1 d–f require 30 minutes and should be followed by overnight drying. Parts I g and Parts II h–k require 30 minutes.

MATERIALS NEEDED

per student

cupric sulfate, anhydrous, 0.1 g
 cupric sulfate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$), 10 g
 ammonia solution, 10 ml, 15 *M* (conc. reagent)
 ammonia solution, 5 ml, 6 *M*, (40 ml conc./100 ml)
 ammonia-alcohol mixture, 8 ml
 (500 ml conc. $\text{NH}_3(\text{aq})$ + 500 ml ethyl alcohol)
 ethyl alcohol, 18 ml

beaker, 100 ml
 funnel and filter paper to fit
 graduated cylinder, 10 ml
 evaporating dish
 watch glass to fit evaporating dish
 1 stirring rod
 3 test tubes, 13 × 100 mm

per class

15 balances, centigram
15 mortars and pestles

10 squeeze bottles of distilled water
10 aspirators and hoses

PRELAB DISCUSSION

None is needed.

PRECAUTIONS

Concentrated $\text{NH}_3(\text{aq})$ is hard on eyes and nostrils. Use adequate ventilation. Students should wear safety glasses and aprons.

POSTLAB DISCUSSION

The following information can be used to develop many points.

PART I

- b. The $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$ should be pulverized for more rapid solution.
- c. If the alcohol mixes too quickly, the solid will be a mass of tiny crystals that are slow to settle and difficult to separate from the liquid.
- f. Drying overnight will ensure more complete removal of excess water.
- g. 0.02 mole of this complex (molar mass = 246) is 4.92 grams. A yield of 80% is reasonable. 0.08 mole of ammonia would have been used.

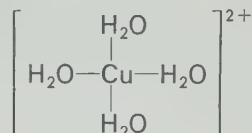
PART II

- h. The blue color typical of the aquated Cu^{2+} ion is seen when water is added to the anhydrous salt. When ammonia solution is added, a deep blue color is produced. If $\text{NH}_3(\text{aq})$ is added too rapidly the student may see the interim production of white copper hydroxide before the complex is formed.
- i. A royal blue color is seen. Upon dilution, the solution may become milky and lose the deep blue color. Whether this happens depends upon the amount of salt used, and, since the amount is not specified, different students will undoubtedly get different results. A reasonable explanation is that the $\text{Cu}(\text{H}_2\text{O})_4^{2+}(\text{aq})$ ions are too pale in color to be seen when diluted and that the $\text{Cu}(\text{NH}_3)_4^{2+}(\text{aq})$ ions change to $\text{Cu}(\text{H}_2\text{O})_4^{2+}(\text{aq})$ upon addition of more water. The milkiness that may be observed is probably $\text{Cu}(\text{OH})_2(\text{s})$.
- j. The complex salt decomposes very easily to leave a red-brown solid. The odor of ammonia is clearly evident, as is that of SO_2 (see Question 3 for equation). $\text{H}_2\text{O}(\text{g})$ is also given off.
- k. The crystals become gray in color and $\text{H}_2\text{O}(\text{g})$ is given off. Severe heating produces Cu_2O , CuO , and $\text{SO}_2(\text{g})$.

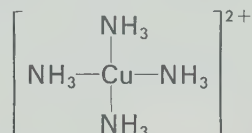
RESULTS OF PROCESSING THE DATA

- 1) In Part II h, account for the successive color changes noted in terms of the structure of the ion containing copper.

The first aqua blue will be attributed to aquated copper(II) ions,



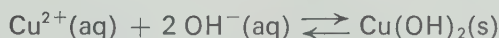
although the ions in the solution are not necessarily those in the hydrate. The deeper blue is due to the formation of tetraamminecopper(II) ions.



These ions are also aquated, although the water is seldom shown in the formula.

- 2) What are the ionic species present when the complex salt tetraamminecopper(II) sulfate is dissolved in a small quantity of water? Give a reasonable explanation of the changes which took place as more water was added.

The ionic species present in the crystal are $\text{Cu}(\text{NH}_3)_4^{2+}$ and $\text{SO}_4(\text{H}_2\text{O})^{2-}$. In water these become further aquated. The changes in color as water is added are due, at first, to aquation and, as still more is added, to H_2O replacement of NH_3 in association with the copper. The reaction



shifts the equilibrium of the reaction



to the right; as a result, less NH_3 is available for complex ion formation. Thus as more $\text{Cu}(\text{OH})_2(\text{s})$ is formed, more $\text{NH}_4^{+}(\text{aq})$ will also be formed.

- 3) Account for the changes you noted when each salt was heated in Parts II j and k.

When the tetraamminecopper(II) sulfate is heated the student will notice some condensation of moisture and the strong odor of ammonia. He may check for ammonia by holding moist red litmus at the mouth of the test tube. Even with gentle heating he will note that the solid becomes red or black. In addition to the odor of ammonia he will detect the strong odor of SO_2 . The odor of NO_2 would be obscured; if only a little is produced the color might not be noticeable. The red solid is probably copper(I) oxide and the black one, copper(II) oxide. A suggested equation is



The NO will react with O_2 in the air to form NO_2 .

- 4) Why was a yield of 0.02 mole expected for the salt you prepared in Part I?

Since 0.02 mole of the single salt was used, the yield for the complex salt would be 0.02 mole. The equation is:



Experiment 45

Preparation of Potassium Dichromate

PURPOSE AND SUMMARY

To make use of oxidation-reduction reactions in an inorganic preparation and to illustrate some of the reactions of a transition metal.

The student prepares potassium dichromate *via* a three step conversion of $\text{Cr}(\text{H}_2\text{O})_6^{3+}$ to $\text{Cr}(\text{H}_2\text{O})_2(\text{OH})_4^-$ to CrO_4^{2-} to $\text{Cr}_2\text{O}_7^{2-}$, using an excess of strong base, an excess of hydrogen peroxide, and an excess of concentrated acetic acid.

TIMING

This experiment can be done anytime after Section 20-5.

There are convenient stopping places. Steps a–d require about 30 minutes, with an additional 10 to 15 minutes required to reduce the volume to about 30 ml. About 15 minutes are required to do steps e and f. An additional 10 minutes are required for step g. The final weighing of the crystals takes just a few minutes.

MATERIALS NEEDED

per student or pair

- 1 conical flask, 250 ml
- 1 beaker, 100 ml
- 1 ring stand, iron ring, gauze, and burner or hot plate
- 1 medicine dropper
- 1 or 2 boiling chips
- 1 balance, centigram
- ice in insulated container, about 8–10 cubes
- 2.5 grams chromium(III) acetate. See Lab Hint 1.
- KOH, 25 ml, 6 *M*, (33.6 grams/100 ml solution). See Lab Hint 2.
- H_2O_2 3% solution, 50 to 60 ml
- acetic acid, 3 ml, 18*M*
- ethanol, 10 ml

LAB HINTS

1. Chromium(III) acetate is sold as $\text{Cr}(\text{CH}_3\text{COO})_3 \cdot x \text{H}_2\text{O}$, with an assay of 23–25% Cr. This indicates an average of about one molecule of H_2O for each molecule of $\text{Cr}(\text{CH}_3\text{COO})_3$. The monohydrate formula is given in the Laboratory Manual.
2. Since KOH absorbs CO_2 readily, a freshly prepared solution should be used in this experiment.
3. In step d the excess of H_2O_2 is boiled off. If it is not, Cr^{3+} is formed instead of Cr^{6+} when acid is added. If the solution turns green upon addition of acid, have the students add more KOH and H_2O_2 , and reheat for 10 minutes. The solution must be *gently* boiled, since there is danger of spattering, even when a 250-ml conical flask is used. But if an overnight period is available, the solution may be concentrated by heating in a drying oven at 30–50°C.

PRELAB DISCUSSION

Remind the students of their previous experience observing reactions involving $\text{Cr}_2\text{O}_7^{2-}$, CrO_4^{2-} , and Cr^{3+} . Have them review their observations from Experiments 30 and 37.

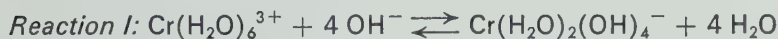
PRECAUTIONS

Chromium compounds are toxic. Hydrogen peroxide can be dangerous. With 3% H_2O_2 there is no danger. Higher concentrations cause severe burns. If higher concentrations are to be diluted by the student, he should wear safety glasses. Glasses should also be worn when handling glacial acetic acid. Caution student to use boiling chips and boil the solution *gently* in step d, to avoid spattering.

RESULTS OF PROCESSING THE DATA

- 1) Write the equation for each of the reactions involved in the preparation of potassium dichromate. Show your calculations for the amount of hydrogen peroxide required and for the expected yield of potassium dichromate.

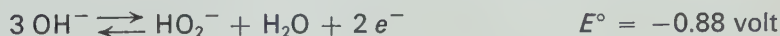
The balanced equations are:



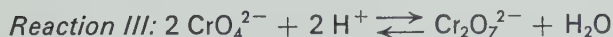
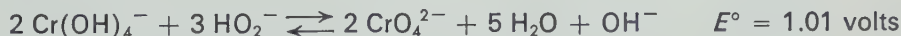
Reaction II: One half-reaction is



A footnote informs the student that $\text{Cr}(\text{OH})_4^-$ is the same as $\text{Cr}(\text{H}_2\text{O})_2(\text{OH})_4^-$, shown in the introduction, except that the water of aquation is omitted. The other half-reaction is



The net equation is



For 0.01 mole of chromium acetate, 0.015 mole of H_2O_2 will be needed, since 2 moles of $\text{Cr}(\text{OH})_4^-$ require 3 HO_2^- (*Reaction II*).

$$(0.015 \text{ mole})(34 \text{ g/mole}) = 0.51 \text{ g}$$

Since a 3% H_2O_2 solution contain 0.03 g $\text{H}_2\text{O}_2/\text{ml}$,

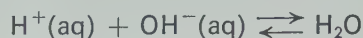
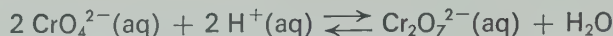
$$\frac{0.51 \text{ g}}{0.03 \text{ g/ml}} = 17 \text{ ml}$$

Each mole of chromium acetate yields $\frac{1}{2}$ mole of $\text{K}_2\text{Cr}_2\text{O}_7$, hence 0.01 mole of chromium acetate is produced from 0.005 mole of dichromate. This is

$$(0.005 \text{ mole})(294 \text{ g/mole}) = 1.47 \text{ g}$$

- 2) What ionic species were present in the final acidic solution before it was cooled? What equilibria are established?

The ionic species were $\text{CH}_3\text{COO}^-(\text{aq})$, $\text{H}^+(\text{aq})$, $\text{K}^+(\text{aq})$, $\text{Cr}_2\text{O}_7^{2-}(\text{aq})$, and a small amount of $\text{CrO}_4^{2-}(\text{aq})$;



In both these equilibrium reactions the concentration of products is greater than the concentration of reactants.

- 3) Considering the following solubility data, would you expect your sample of potassium dichromate to be relatively pure? Why?

Temperature, °C	Solubility in g/100 of water	
	KCH_3COO	$\text{K}_2\text{Cr}_2\text{O}_7$
0	217	5
20	256	12
40	323	26
60	350	43
80	380	61
100	—	80

The sample should be quite pure. KCH_3COO is quite soluble and will stay in solution. The data for the solubilities of these two compounds gives the following curves:

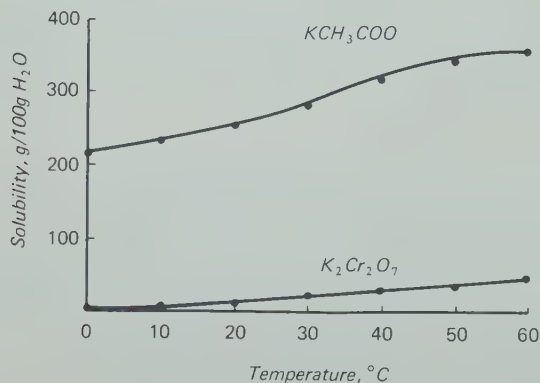


Figure 33

At 10°C almost 2 grams of $\text{K}_2\text{Cr}_2\text{O}_7$ would dissolve in 25 ml water. In this experiment 0.005 mole of $\text{K}_2\text{Cr}_2\text{O}_7$ may be produced, which is 1.5 grams (0.005 mole $\times$ 294 g/mole). However, only 0.01 mole of K^+ is required and 0.15 mole was added (0.025 liter $\times$ 6 moles/liter). This large excess of K^+ will result in the crystallization of considerable $\text{K}_2\text{Cr}_2\text{O}_7$ from this solution.

At 10°C the K_{sp} of $\text{K}_2\text{Cr}_2\text{O}_7 = \sim 5 \times 10^{-2}$. If the concentration of K^+ is 0.15 mole in 25 ml, or 6 M, the amount of $\text{Cr}_2\text{O}_7^{2-}$ remaining in the solution is only about 0.01 gram. This is consistent with the percent yield (nearly 100%) actually obtained in two trial runs of this experiment.

- 4) Compare the E° values of the half-reactions for the oxidation of Cr(III) to Cr(VI) in a basic solution with that for the oxidation in an acidic solution. See the introductory section of this experiment for the former value and Appendix 4 for the latter value. Predict which oxidation will favor products more at equilibrium.

The reaction favors products in basic solution, whereas in the acidic solutions, reactants are favored. For equations in basic solution, see *Reaction II* above.

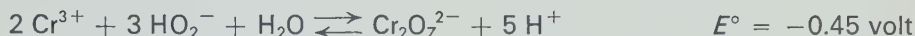


The products are favored.

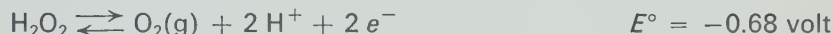
In acid solution



Therefore



The reactants are favored. In fact, with H_2O_2 in acid solution,



hence the reaction would be



rather than the oxidation of Cr^{3+} to Cr^{6+} .

- 5) Note the half-reaction for the oxidation of H_2O_2 for which the E° value is -0.68 volt (Appendix 4). Can you now suggest a reason for decomposing the excess H_2O_2 in step d before the solution was acidified to convert chromate ions to dichromate ions?

See the answer to 4). If excess H_2O_2 were left, the CrO_4^{2-} or $\text{Cr}_2\text{O}_7^{2-}$ would be reduced to Cr^{3+} when the solution was acidified.

- 6) Is the conversion of chromate ion to dichromate ion an oxidation reaction? Explain.

In both CrO_4^{2-} and $\text{Cr}_2\text{O}_7^{2-}$ the oxidation number of Cr is $+6$, hence this conversion is not an oxidation-reduction reaction.

Experiment 46

Preparation of Chrome Alum

PURPOSE AND SUMMARY

To give the student further experience with oxidation-reduction reactions and with crystallization in an inorganic preparation.

The student writes a balanced equation representing the reduction of potassium dichromate using sulfur dioxide in acid solution. From the balanced equation he calculates the moles of $\text{Na}_2\text{S}_2\text{O}_5$ required to effect the reduction. After the reduction is complete the saturated solution

of chrome alum, $\text{KCr}(\text{SO}_4)_2 \cdot 12 \text{H}_2\text{O}$, is allowed to produce crystals slowly as it cools and evaporates over a period of several days.

TIMING

Parts of three periods are required. Step a requires about 10 minutes, steps b–g about 45 minutes, and step h about 10 minutes.

This experiment fits best anytime after Section 20-5 in the Textbook.

MATERIALS NEEDED

per student or pair

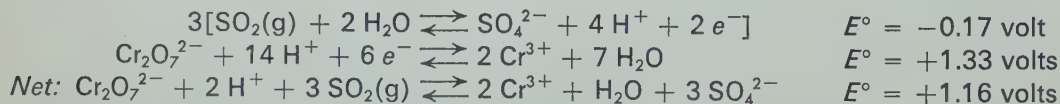
2 conical flasks, 250 ml	1 filter paper
1 evaporating dish and watch glass	1 funnel and funnel rack
1 delivery tube, 30–40 cm, 6 mm I.D.	ice in insulated container, about 8–10 cubes
1 glass bend and glass tubing, each about 6 mm O.D. $\times$ 10 cm	1 graduated cylinder, 25 ml
1 #6 two-hole stopper, rubber	1 balance, centigram
1 thistle or funnel tube	potassium dichromate, 15 grams
1 ring stand, iron ring, gauze, and clamp	sodium metabisulfite ($\text{Na}_2\text{S}_2\text{O}_5$), 30 grams
1 burner	sulfuric acid, 30 ml, 6 <i>M</i> (340 ml conc. reagent/liter solution)
1 thermometer, -20° to 150°C	sulfuric acid, 60 ml, 3 <i>M</i> (170 ml conc. reagent/liter solution)
1 medicine dropper	

LAB HINTS

None.

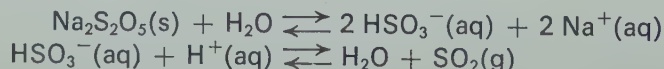
PRELAB DISCUSSION

Before coming to the laboratory, the student should have worked out the following equations and amounts of reactants:

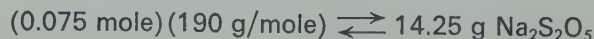


step a: 0.05 mole of $\text{K}_2\text{Cr}_2\text{O}_7$ is (0.05 mole) (294 g/mole) = 14.7 grams.

step b: From the equations above we see that three moles of SO_2 are needed per mole of $\text{K}_2\text{Cr}_2\text{O}_7$, hence 0.15 mole SO_2 is needed for 0.05 mole $\text{K}_2\text{Cr}_2\text{O}_7$. The equations for the production of SO_2 are



Therefore one mole of $\text{Na}_2\text{S}_2\text{O}_5$ will produce two moles of SO_2 , and since 0.15 mole SO_2 is needed, 0.075 mole of $\text{Na}_2\text{S}_2\text{O}_5$ will be needed.



The student should use about 30 grams of $\text{Na}_2\text{S}_2\text{O}_5$ because the yield is not 100%.

When heated, the hydrated, violet chromium sulfate, $[\text{Cr}(\text{H}_2\text{O})_6]_2(\text{SO}_4)_3 \cdot n \text{H}_2\text{O}$, forms green chromium(III) sulfates called sulfato-complex compounds. Most of these complex compounds have a very poor ability to crystallize. It is, therefore, extremely important that the temperature be controlled here to prevent the formation of these troublesome compounds, which tend to inhibit crystallization of the chrome alum.

step e-f: Two moles of $\text{H}^+(\text{aq})$ are required for one mole of $\text{Na}_2\text{S}_2\text{O}_5$, therefore 0.15 mole will be required for 0.075 mole. Since 6 *M* H_2SO_4 is being used, there are available two H^+ per mole, or 0.012 mole H^+/ml . To obtain 0.15 mole of H^+ we need $0.15 \text{ mole} / 0.012 \text{ mole/ml} = 12.5 \text{ ml}$. Thus 12 to 13 ml of H_2SO_4 would be sufficient, but owing to dilution, slightly more than this will be needed. Two 10 ml portions should be sufficient.

The student will need to watch this reaction carefully. Very little heating of the generator is needed to maintain the production of SO_2 so that it will enter the solution only as fast as it can react. If allowed to cool too much, however, liquid from the reaction flask will be forced back. The time needed will vary considerably, but 30 minutes seems a good average. The test with a drop on some filter paper works very well and is clear-cut.

step g: It may occur to the student to reduce the volume somewhat here, but if he does, the solution will become quite viscous and form a syrupy mixture which will not crystallize, therefore we suggest that he be told not to do this. It is probably best to assign this experiment far enough from the end of the semester so that the crystals have plenty of time to develop; otherwise you will have to be satisfied with very small ones. Since the solution contains enough acid to react with string, we advise against suspending a string on which crystals may grow.

step h: The maximum possible yield is 0.1 mole, since one mole of $\text{K}_2\text{Cr}_2\text{O}_7$ will produce two moles of chrome alum if one considers the ionic species present in the crystal lattice to be $\text{Cr}(\text{H}_2\text{O})_6^{3+}$, $\text{K}(\text{H}_2\text{O})_6^+$ and SO_4^{2-} . Thus $(0.1 \text{ mole})(499 \text{ g/mole}) = 50 \text{ grams}$. At room temperature 24 grams of chrome alum dissolve in 100 grams of water. When poured into the evaporating dish, there will be about 50–60 ml of solution, so one may expect to obtain 30–35 grams of crystals. There is, however, a great tendency for a supersaturated solution to form, and the actual yield is more likely to be 20–25 grams.

PRECAUTIONS

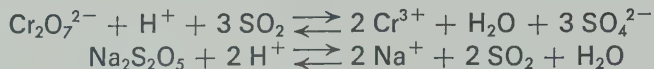
Emphasize the importance of handling sulfuric acid correctly and of thorough cleanup if any is spilled. The generation of SO_2 must be carried on in a well-ventilated room or under a hood. Take care that the gas is thoroughly reacted with the solution through which it is being bubbled and that it is not produced too rapidly. When disposing of the generator after the reaction is complete, do not breathe the fumes. Wear safety glasses and aprons.

POSTLAB DISCUSSION

Discuss questions and exercises under *Processing the Data*.

RESULTS OF PROCESSING THE DATA

The equations for the reactions involved in the experiment as requested in the introduction are given below:

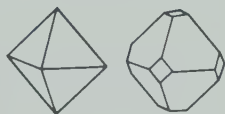


1. Under what conditions could you grow large crystals of chrome alum?

The student will suggest slow evaporation or gradual cooling to get large crystals. He may know about the method of "seeding" with a small well-formed crystal. If any students are particularly interested, a good reference is the paperback book by Holden and Singer, *Crystals and Crystal Growing*, Doubleday (an Anchor Book) (1960).

2. Examine one of the larger crystals you prepared. Note any outstanding features of its geometric symmetry, and prepare a sketch showing them.

He may observe that the crystal is octahedral. He will certainly note that most of the crystals show a distinct triangular face, but some rectangular faces. Many will be pyramids.



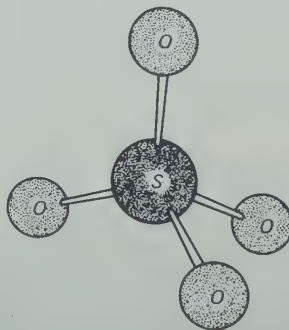
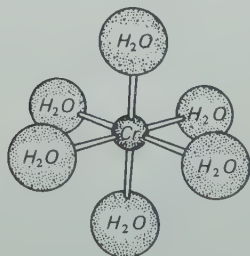
"Tablet"
growing on
bottom of
container



"Sketch" of
crystals
produced on
trial run of
experiment

3. What are the building blocks of the chrome alum crystal? Draw a three-dimensional structural formula for each of the ionic species involved.

The building blocks of the crystal are Cr^{3+} ions, each hydrated with six molecules of water, similarly hydrated potassium ions, again with six molecules of H_2O , and SO_4^{2-} ions.



4. Show calculations of the amounts of reactants required and the theoretical yield expected.

See the discussion of steps a for amount of $\text{Cr}(\text{H}_2\text{O})_6^{3+}$, b for amount of $\text{K}_2\text{Cr}_2\text{O}_7$, c for amount of $\text{Na}_2\text{S}_2\text{O}_5$, d-e for amount of H_2SO_4 , and h for theoretical yield.

Chemicals and Equipment Used

The following list is divided into three main categories. **Supplies** consists of materials and chemicals consumed during the school year. **Equipment** includes reusable items—glassware and hardware. **Miscellaneous** items are those which do not normally fall into the above categories. The supplies and equipment are not much different from what is normally stocked.

The list indicates the amounts needed for a class of 30 students who are doing all the experiments. It is assumed that the students will work in groups or in pairs as recommended in the individual experiment guides. Material for Demonstrations is included. Chemicals are to be analytical grade unless otherwise specified. A modest surplus has been allowed. It is advisable to check individual guides to experiments before deciding to purchase supplies. Some substitutions may be possible. Should you wish to order material, the catalog numbers shown are those of Macalaster Scientific, 186 Third Avenue, Waltham, Mass. 02154.

Supplies (expended during school year)

CHEMICALS

MSC No.	MSC Pkg. Qty.	Compound	Purchased As	Quantity	Used in (<i>Note 1</i>)
79032	1 lb	Acetamide		150 g	15
79302	1 pt	Acetic acid	glacial	1200 ml	D23, 24, 26, 30, 31, 33, 40, 45
57040	1 pt	Acetone		4 pt	7, D11, 36
57111	1/4 lb	Agar	powder	60 g	35
25021	25 g	Alizarin Yellow R		0.1 g	33
1035	12" × 25'				
	roll	Aluminum foil	household wrap	8 ft ²	2
25022	1/4 lb	Aluminum chloride	AlCl ₃ ·6 H ₂ O	6 g	17
25024	1 lb	Aluminum sulfate	Al ₂ (SO ₄) ₃ ·18 H ₂ O	20 g	17
79286	3 1/2 oz	Ammonia, aqueous	conc. reagent	800 ml	D23, 20, 22, 23, 30, 31, 44
25029	1 lb	Ammonium carbonate		200 g	D21, 20, 37
30407	1/4 lb	Ammonium chloride		35 g	D21, 24
25033	1/4 lb	Ammonium oxalate	(NH ₄) ₂ C ₂ O ₄ ·H ₂ O	30 g	20
79287	1 lb	Ammonium peroxydisulfate	(NH ₄) ₂ S ₂ O ₈	46 g	28

Note 1—Demonstrations are shown by *D* before a number.

MSC No.	MSC Pkg. Qty.	Compound	Purchased As	Quantity	Used in
57215	1/4 lb	Ammonium sulfate		138 g	20
25034	1/4 lb	Ammonium thiocyanate		3 g	D31, 43
57271	1/4 lb	Barium chloride	BaCl ₂ ·2 H ₂ O	30 g	9, 17
25039	1 lb	Barium hydroxide	Ba(OH) ₂ ·8 H ₂ O	50 g	17, D23, D31, 37
25040	1/4 lb	Barium nitrate		50 g	17, 19, 20, 23, 30
57298	1 pt	Benzene		150 g	D22, 39
25049	1/4 lb	Bromine		2 g	34, 39
57365	1 pt	1-Butanol	<i>n</i> -butyl alcohol	150 ml	39
25052	1 kg	2-Butanol	2-methyl- 1-propanol	150 ml	39
25053	1 pt	<i>t</i> -Butanol	<i>tert</i> -butyl alcohol	150 ml	39
57400	1 lb	Calcium carbonate	marble chips	40 g	24
25058	1 lb	Calcium chloride	anhydrous, granular	20 g	15, D21, D23
25059	1 lb	Drierite	#12 mesh	100 g	9, 40
57410	1 lb	Calcium hydroxide	powder, tech.	6 g	D23, 30, 33
25061	1/4 lb	Calcium nitrate	Ca(NO ₃) ₂ ·4 H ₂ O	24 g	20
57420	1 lb	Calcium sulfate	CaSO ₄ ·2 H ₂ O	20 g	9
79285	250 g	Camphor		10 g	15
—	—	Chlorine	bottled	1 l	34
25068	1/4 lb	Chromium(III) acetate	Cr(CH ₃ COO) ₃ ·H ₂ O 23–25% Cr	90 g	45
25070	4 oz	Chromium sulfate	Cr(SO ₄) ₃ ·H ₂ O 65% Cr(SO ₄) ₃	60 g	37
30412	1/4 lb	Citric acid	monohydrate	1 g	33
57490	1/4 lb	Cobalt(II) chloride	CoCl ₂ ·6 H ₂ O	31 g	17, 43
25072	1/4 lb	Cobalt(II) nitrate	Co(NO ₃) ₂ ·6 H ₂ O	20 g	17
79288	1/4 lb				
	spool	Copper wire	B and S, gauge 22	1/4 lb	11, D14, 39
79289	1 lb				
	spool		B and S, gauge 16	1 lb	7, D14, 35, 36
79290	1	Copper screen	20 mesh	1500 cm ²	36
79291	1	Copper sheet	B and S, gauge 20 to 30	750 cm ²	34
25073	1/4 lb	Copper(II) chloride	CuCl ₂ ·2 H ₂ O	330 g	2, D21
25075	1/4 lb	Copper(II) nitrate	Cu(NO ₃) ₂ ·3 H ₂ O	120 g	34
25076	1 lb	Copper(II) sulfate	CuSO ₄ ·5 H ₂ O	350 g	6, 9, 28, 36, 44
25077	1/4 lb	Copper(II) sulfate	anhydrous	3 g	44
25078	1 pt	Cyclohexane		30 ml	39
25079	250 g	Cyclohexene		30 ml	39
25083	1 oz	Dimethylglyoxime		1 g	43
25085	1 pt	Ethanol	alcohol, denatured	1500 ml	30, 39, 40, 43, 44, 45
57700	1/4 lb	Ferric chloride	FeCl ₃ ·6 H ₂ O	40 g	17, D29, 37, 41, 43

MSC No.	MSC Pkg. Qty.	Compound	Purchased As	Quantity	Used in
25088	1/4 lb	Ferric nitrate	$\text{Fe}(\text{NO}_3)_3 \cdot 9 \text{H}_2\text{O}$	120 g	D29, 29
57735	1/4 lb	Ferrous sulfate	$\text{FeSO}_4 \cdot 7 \text{H}_2\text{O}$	60 g	24, 35, 37
57855	1 pt	Hydrochloric acid	12 M	3500 ml	11, D23, 20, 22, 24, 26, 27, 30, 31, 32, 33, 35, 37, 39, 43
—	—	Hydrogen chloride gas	bottled	1 l	D22
57870	1 pt	Hydrogen peroxide	3% solution	3 pt	37, 42, 45
25095	10 g	Indigo Carmine		0.25 g	33
57925	1 oz	Iodine		35 g	34, 42
33412	1 lb	Iron filings	fine mesh	60 g	6
46576	1 pkg	Iron nails	6–8 penny	270	35
79292	1	Lead	sheet, 1/32"	1500 cm ²	34, 36
25105	4 oz	Lead dioxide		30 g	24
30436	1/4 lb	Lead nitrate		50 g	16, D20, 22, D27, 34, 37
30421	1 roll	Magnesium	ribbon, 1 oz roll	6 g	11, 27
58190	1/4 lb	Magnesium chloride	$\text{MgCl}_2 \cdot 6 \text{H}_2\text{O}$	10 g	17
25110	1 lb	Magnesium nitrate	$\text{Mg}(\text{NO}_3)_2 \cdot 6 \text{H}_2\text{O}$	50 g	20, 37
25111	1/4 lb	Magnesium oxide		30 g	27, 33
25113	1 lb	Magnesium sulfate	$\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$	30 g	9, 17, 19
25116	4 oz	Manganese dioxide		5 g	D18
25118	1/4 lb	Manganous sulfate	$\text{MnSO}_4 \cdot \text{H}_2\text{O}$	35 g	9, 24
25119	1/4 lb	Mercury		1/4 lb	10
25122	1 oz	Mercurous nitrate	$\text{Hg}_2(\text{NO}_3)_2 \cdot 1 \text{H}_2\text{O}$	6 g	22
72	1 pt	Methanol		250 ml	39, 40
25127	1 oz	Methyl Orange		1 g	33
25132	1/4 lb	Nickel(II) chloride	$\text{NiCl}_2 \cdot 6 \text{H}_2\text{O}$	5 g	17, 43
58330	1 lb	Nitric acid	15 M	350 ml	8, 22, 23, 30, 31, 35, 42
25136	25 g	Orange IV		0.1 g	33
25137	1 lb	Oxalic acid	$\text{H}_2\text{C}_2\text{O}_4 \cdot 2 \text{H}_2\text{O}$	1 g	33
—	—	Oxygen	bottled	30 l	5
30426	1 lb	Paradichlorobenzene	moth flakes	450 g	12
58445	1/4 lb	Phenolphthalein		2 g	24, 33, 35, 41
25142	1 pt	Phosphoric acid		0.003 mole	D23
25146	1/4 lb	Potassium bromide		24 g	24, 37
25147	1 lb	Potassium carbonate		20 g	9
25149	1/4 lb	Potassium chloride		5 g	17
25150	1/4 lb	Potassium chromate		150 g	17, 19, 20, 22, 24, 30
58742	1/4 lb	Potassium dichromate		500 g	30, 37, 46
25152	1/4 lb	Potassium ferricyanide	$\text{K}_3\text{Fe}(\text{CN})_6$	4 g	35
25153	1/4 lb	Potassium ferrocyanide	$\text{K}_4\text{Fe}(\text{CN})_6 \cdot 3 \text{H}_2\text{O}$	10 g	19
79293	1 lb	Potassium hydrogen oxalate (or sodium)		20 g	32

MSC No.	MSC Pkg. Qty.	Compound	Purchased As	Quantity	Used in
25155	1/4 lb	Potassium hydrogen phthalate (or sodium)		20 g	32, 33
25156	1 lb	Potassium hydrogen sulfate		20 g	32, D23
79294	1 lb	Potassium hydrogen tartrate		21 g	32, 33
25158	1 lb	Potassium hydroxide		400 g	17, D23, 30, 31, 35, D32, 42, 45
25159	1/4 lb	Potassium iodate		50 g	42
25160	1/4 lb	Potassium iodide		200 g	15, 28, 37, 41, 42
58483	1/4 lb	Potassium nitrate		1 g	35
30425	1/4 lb	Potassium permanganate		25 g	24, 37, 39
79295	1 lb	Potassium sulfate		0.5 g	D23
25163	1/4 lb	Potassium thiocyanate		2 g	D29, 29, 43
79296	1 lb	Resin, ion exchange	Dowex AG2-X8	120 g	43, (reusable)
79297	1/4 lb	Salicylic acid		100 g	40
25169	4 oz	Silver chloride		75 g	36, (reclaim from Expt. 8)
25170	1/4 lb	Silver nitrate		300 g	7, 17, D21, D22, D23, 19, 22, 23, 24, 42
25171	1/4 lb	Sodium		3 g	D14, 39
25172	1/4 lb	Sodium acetate	$\text{NaCH}_3\text{COO} \cdot 3 \text{H}_2\text{O}$	500 g	18, 24, 33, 36, 37
25173	1 lb	Sodium bromide		3 g	34
25175	1 lb	Sodium carbonate	$\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$	100 g	9, 23, 33, 35, 40
25177	1/4 lb	Sodium chloride		100 g	8, D21, D22, D23, 19, 23, 24, 34, 35, 37
25178	1 lb	Sodium chromate		5 g	17
25179	1 lb	Sodium dichromate		3 g	35
25182	1 lb	Sodium hydrogen carbonate	anhydrous	1 g	D21, 24, 33
25185	1 lb	Sodium hydroxide	pellets	500 g	17, D23, 24, 26, 30, 31, 32, 33, 35, 37, 39
25186	1 qt	Sodium hypochlorite	5% commercial bleach	10 ml	42
25187	1/4 lb	Sodium iodide		70 g	D18, 15, 16, D20, 23, 24, 34
25235	1 lb	Sodium metabisulfite (sodium hydrogen sulfite)	$\text{Na}_2\text{S}_2\text{O}_5$	1000 g	36, 42, 46
25188	1 lb	Sodium monohydrogen phosphate (dibasic)		1 g	D29
25189	1/4 lb	Sodium nitrate		8 g	17
25190	1/4 lb	Sodium oxalate		15 g	24, 35
25192	1/4 lb	Sodium phosphate, tribasic	$\text{Na}_3\text{PO}_4 \cdot 12 \text{H}_2\text{O}$	5 g	33, 35
25193	1 lb	Sodium sulfate	anhydrous	40 g	17, D21, 19, 23, 36, 37
25195	1/4 lb	Sodium sulfide	$\text{Na}_2\text{S} \cdot 9 \text{H}_2\text{O}$	3 g	37
25196	1 lb	Sodium sulfite	anhydrous	25 g	24, 37

MSC No.	MSC Pkg. Qty.	Compound	Purchased As	Quantity	Used In
25198	1 lb	Sodium thiocyanate		1 g	D29, 35
25199	1 lb	Sodium thiosulfate	photo grade		
			hydrated	800 g	7, 14, 28, 36
25200	$\frac{1}{4}$ lb	Starch	soluble	5 g	28, 42
25204	$\frac{1}{4}$ lb	Strontium nitrate		30 g	17, 20
58740	1 lb	Sugar, sucrose		10 g	D21
58750	1 pt	Sulfuric acid	18 M	1250 ml	D18, D23, 24, 30, 31, 35, 36, 37, 39, 40, 42, 46
79298	500 ml	Trichlorotrifluoroethane (TTE)	Dupont TF solvent	350 ml	34, 37, 39, 41
58885	1 lb	Urea	NH_2CONH_2	10 g	15
79299	1	Zinc	sheet, $\frac{1}{16}$ "	750 cm ²	34, 35
79300	$\frac{1}{4}$ lb	Zinc nitrate	$\text{Zn}(\text{NO}_3)_2 \cdot 6 \text{H}_2\text{O}$	150 g	19, 34
58995	1 lb	Zinc sulfate	$\text{ZnSO}_4 \cdot 7 \text{H}_2\text{O}$	50 g	9, 37

Equipment (not expended during the school year)

GLASSWARE

MSC No.	MSC Pkg. Qty.	Item	Quantity	Used in
54196	12	Beaker, 50 ml	20	D21, D23
54197	12	100 ml	60	2, 6, 7, 8, 16, 24, 28, 29, 31, 44, 45
54199	12	250 ml	30	4, 7, 13, 18, 22, 24, 26, 35, 40
54200	12	400 ml	30	11, 12, 36
54204	1	2000 ml	3	D11
63077	12	Bottles, 4 oz, screw cap	75	2, 18, 24, 26, 27
79269	1	2 liter	12	26, 27, 28, 31
20757	10	Buret, 25 ml	60	16, 32, 33
20423	1	Condenser, small	30	40
30272	1	Crucible	30	9
79256	1	Crucible cover	30	9
79257	1	Cylinder, hydrometer	10	11
54270	12	Dropper bottles, 2 oz	3 gross	17, 19, 20, 22, 23, 30, 33, 37, 39, 42
20392	1	Evaporating dish, 75 mm	30	44, 46
54886	12	Flask, conical, 250 ml	30	D2, D17, 32, 33, 45, 46
		Funnel, 75 mm dia.		
54970	1	short stem	30	8, 16, 42, 44, 46
20397	1	thistle tube	15	46
20367	1	separatory, 25 ml	15	40

MSC No.	MSC Pkg. Qty.	Item	Quantity	Used in
79258	1	Gas measuring tube, 50 ml	30	11
56320	12	Glass stirring rod 6 mm × 20 cm	30	frequent use
79284	30	Glass tubing capillary, 1.0 mm O.D. × 20 cm	30	10
79259	8	6 mm	30 ft	43, 46
79260	1	10 mm × 60 cm	30	40
79261	1	12 mm × 30 cm	15	43
		Graduated cylinders		
20704	10	10 ml	30	frequent use
20707	10	25 ml	30	3, 4, 7, 8, 11, 28, 29, 46
20710	10	50 ml	30	16, 31
20713	10	100 ml	30	13, 14, 26, 27
20716	10	250 ml	8	5
20719	10	500 ml	8	25
79262	1	Jar, half gallon	15	5 (empty acid bottles)
54765	12	Medicine dropper	30	5, 29, 33, 41, 45, 46
55673	12	Mortar and pestle	15	44
54690	100	Petri dishes, covers unnecessary	60	35
		Test tubes, hard glass		
20321	72	13 × 100 mm	330	16, 20, 22, 24, 30, 33, 34, 35, 37, 39, 40, 42, 44
20322	72	18 × 150 mm	120	7, 14, 15, 18, 28, 42
20323	12	25 × 200 mm	30	10, 40
20406	1	U-tube, 150 mm	8	37, 41
20404	30	Vial, flat bottom 12 ml	150	29
56984	12	Watch glass, 75 mm dia.	30	7, 44, 46

Equipment (not expended during the school year)

HARDWARE

MSC No.	MSC Pkg. Qty.	Item	Quantity	Used in
352	10	Alligator clamps and bell wire leads	40	36, 41
76	1	Ammeter, D.C.	8	36
112	1	Aspirator	10	44, D2, D10

MSC No.	MSC Pkg. Qty.	Item	Quantity	Used in
120	1	Balance, centigram	15	3, 4, 5, 6, 7, 8, D2, 9, 14, 15, 16, 25, 26, 27, 36, 44, 45, 46
79273	1	Barometer	1	11
20351	12	Buret brushes	8	32, 33
54449	12	Burner, Bunsen artificial gas	30	frequent use
54452	12	Burner, Bunsen mixed gas	30	frequent use
54451	12	Burner, Bunsen natural gas	30	frequent use
54540	1	Clamp, buret, double	30	16, 32, 33
320	12	Clamp, utility	30	frequent use
—	—	Clock, sweep-second hand	at least 1	12, 15, 28
—	—	Conductivity apparatus (homemade)	1	D21, D22, D23, D32
2110	1	D.C. source, 12 volts	8	36, 41
55750	1	Drying oven	2	6, 7, 16
54780	1	Electrolysis apparatus	1	Demonstration
20384	1	Funnel rack	15	8, 42, 46
79265	1	Heat lamps, infrared	2	7, 8
79266	1 set	Metal specimens (5 to 10 ml) (See Lab Guide, pp. 6–11.)		3, 4
54551	12	Pinch or screw clamp	30	frequent use
79267	15	Platinum wire, B and S gauge 20–22 (about 5 cm long in glass tube)	15 pcs	20
79230	1	John's apparatus Pressure-temperature apparatus	1	D11
20305	1	Resistor, variable, 18–25 ohms	8	36
56600	2	Ring stand, 18 inch	30	frequent use
56309	1	Ring, iron, 4 inch	30	frequent use
2048	2	Safety glasses	30	frequent use
—	—	Ice chest	1	13, D18, 25, 45
54413	2	Test tube brush	30	frequent use
55120	2	Test tube holder	30	frequent use
56280	2	Test tube rack	30	18, 33, 37, 39
20391	2	Tongs, crucible	30	9, 13
79263	2	Triangle, clay 1½ inch	30	9
54795	1	Water reservoir, pneumatic trough	15	5, 11
55000	2	Wire gauze, 5 × 5 inches with asbestos center	30	frequent use

MISCELLANEOUS

MSC No.	MSC Pkg. Qty.	Item	Quantity	Used in
6210	1 lb	Boiling chips	$\frac{1}{4}$ lb	40, 45
46873	1	Box, shoe or cigar	30	21
79281	1	Can, soup	30	25
79282	1	large fruit juice	30	25
79271	1	lid, 4 inch dia.	30	25
20427	6	Candle, $6 \times \frac{3}{4}$ inch dia. household	30	1, 25
79272	100	Chenille stems, 1 inch long	600	38, D9
79274	1	Electrodes, carbon rods	16	37, 41
—	—	Electrode holder, homemade	16	36
30417	100 sheets/box	Filter paper, 11 cm dia.	300 pcs	8, 16, 42, 44, 46
30435	1 box	Glass wool	$\frac{1}{4}$ lb	43
20377	1 box	Labels, small, gummed	box	frequent use
79275	100 pcs/bottle	Litmus paper, neutral	300 pcs	33, 35
30320	10 boxes	Matches, safety, or flint lighter	30	frequent use
56341	1	Metric rulers, 15 cm, plastic	15	1, 11, 29
56129	2	Pencil, glass marking	30	10, 16, 33, 39
—	—	Plastic squares, thumb protectors	many	16, 18, 24, 30, 34, 37, 39, 41
118	24	(See Expt. 16 this guide.) Plastic bag, 1 qt frozen food	30	5
56350	25 ft	Plastic film, household wrap	30 ft	17, 19, 20, 23, 43
80204	1 roll	Plastic tape	2 rolls	10
2959	1 oz	Rubber band #6	120	5, 10
125	1 oz	Rubber band #8	120	5, 10
66945	50 ft	Rubber tubing, $\frac{1}{4}$ inch ID $\times$ $\frac{1}{16}$ wall	50 ft	5, 46
56560	1 lb	Stopper, rubber #0, 1 hole, 97/lb	30	11
56563	1 lb	#3, 1 hole, 45/lb	30	7
56367	1 lb	#6, 1 hole, 22/lb	30	5
56584	1 lb	#6, 2 hole, 22/lb	30	46
34641	25	Styrofoam, cups 12 oz	30	13, 14
32182	25	8 oz	30	26, 27, 31
20407	25	Styrofoam, spheres $\frac{3}{4}$ inch dia.	200	38
20408	25	1 inch dia.	200	38
20409	25	2 inch dia.	600	38
40674	12	Teaspoons, plastic		2, 18

MSC No.	MSC Pkg. Qty.	Item	Quantity	Used in
79279	1	Thermometers, -20 to 150°C	30	2, 5, 10, 11, 12, 13, 14, 15, 24, 25, 26, 27, 28, 31, 46
80817	1 spool	Thread	30 ft	4
45584	1	Vial, plastic, 7 dram	30	7
30284	1	Wash bottles, polyethylene	15	frequent use
56510	1 M	Wood splints	30	24

List of Film Sources

CHEM Study films may be obtained by purchase, lease, or subscription from any of the offices of Modern Learning Aids shown below. All films obtained by subscription are guaranteed to be delivered on the confirmed date. For further information, write Modern Learning Aids.

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TL 3-1805

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467-6475

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Garfield 1-2516

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Kingsley 5-2500

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16 Spear Street
Yu 2-9414

Seattle, Washington 98103
2100 No. 45th Street
Melrose 3-3878

Washington, D. C. 20006
927 Nineteenth Street, N. W.
Sterling 3-6611

Don Mills, Ont., Canada
(Metro Toronto)
1875 Leslie Street
444-7347

Answers to Exercises, Appendices 6 and 7, Lab Manual

Appendix 6

Expressing Numbers in Exponential Notation

EXERCISES

- 1) a. 4.48×10^6
 b. 3.5×10^6
 c. 0.235×10^5
 d. 810×10^4
 e. 3.245×10^{11}
 f. 2.24×10^{-3}
 g. 4.5×10^{-11}
 h. 0.7616×10^{-2}
 i. 0.0031×10^{-3}
 j. 542.3×10^{-11}
- 2) a. 300 ± 100
 b. $85,000 \pm 1000$
 c. $186,251 \pm 1$
 d. $16,100,000 \pm 100,000$
 e. 0.005 ± 0.001
 f. 0.0000094 ± 0.0000001
 g. 0.000183 ± 0.000001
 h. 0.0155 ± 0.0001
 i. 80.10 ± 0.01
 j. 0.0080 ± 0.0001
- 3) a. 3×10^2
 b. 8.5×10^4
 c. 1.86251×10^5
 d. 1.61×10^7
 e. 5×10^{-3}
 f. 9.4×10^{-6}
 g. 1.83×10^{-4}
 h. 1.55×10^{-2}
 i. 8.0×10^{-3}
 j. 8.010×10^1

Addition and Subtraction

EXERCISES

- 1) 6.9×10^4
- 2) 5.5×10^5
- 3) 9.1×10^{-3}
- 4) 1.09×10^{-2}
- 5) 5.4×10^{-9}
- 6) 8.3×10^7

Multiplication

EXERCISES

- 1) 10^5
- 2) 4×10^{11}
- 3) 9.6×10^{20}
- 4) 1.0×10^6
- 5) 3.9×10^{-10}

Division

EXERCISES

- 1) 10^4
- 2) 9×10^2
- 3) 9×10^{-14}
- 4) 6×10^{10}
- 5) 2.9×10^3

Square Root

EXERCISES

- 1) 10^4
- 2) 5×10^3
- 3) 2.50×10^{-2}
- 4) 4×10^{12}
- 5) 1.3×10^6

Adding, Subtracting, Multiplying, and Dividing Equations

EXERCISES

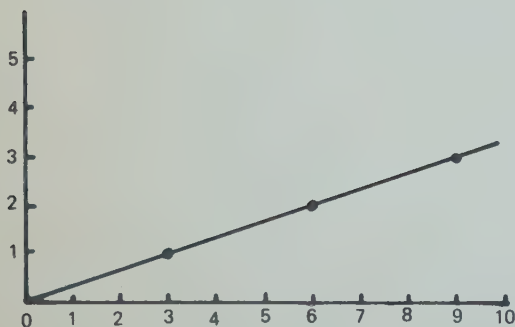
- 1)
$$\begin{array}{r} \text{S}_8(\text{s}) + 8 \text{O}_2(\text{g}) = 8 \text{SO}_2(\text{g}) \\ + 8[\text{SO}_2(\text{g}) + \frac{1}{2} \text{O}_2(\text{g}) = \text{SO}_3(\text{g})] \\ \hline \text{S}_8(\text{s}) + 12 \text{O}_2(\text{g}) = 8 \text{SO}_3(\text{g}) \end{array}$$
- 2)
$$\begin{array}{r} \text{Zn}(\text{s}) = \text{Zn}^{2+}(\text{aq}) + 2 \text{e}^- \\ 2[\text{e}^- + \text{Ag}^+(\text{aq}) = \text{Ag}(\text{s})] \\ \hline \text{Zn}(\text{s}) + 2 \text{Ag}^+(\text{aq}) = \text{Zn}^{2+}(\text{aq}) + 2 \text{Ag}(\text{s}) \end{array}$$
- 3)
$$\begin{array}{r} \text{H}_2(\text{g}) + \frac{1}{2} \text{O}_2(\text{g}) = \text{H}_2\text{O}(\text{g}) + 57.80 \text{ kcal} \\ - [\text{H}_2(\text{g}) + \frac{1}{2} \text{O}_2(\text{g}) = \text{H}_2\text{O}(\text{l}) + 68.32 \text{ kcal}] \\ \hline 10.52 \text{ kcal} + \text{H}_2\text{O}(\text{l}) = \text{H}_2\text{O}(\text{g}) \end{array}$$

Graphing

EXERCISES

1) $x = 3y$

x	y
3	1
6	2
9	3



2) $xy = k$

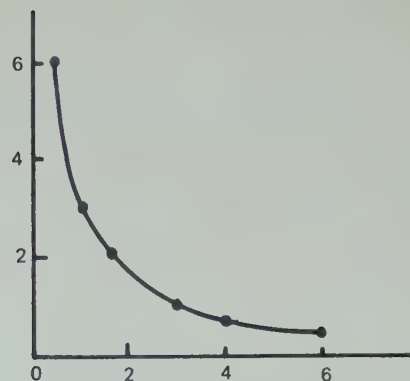
x	y
1	3
3	1

$$x = \frac{k}{y}$$

3	1
---	---

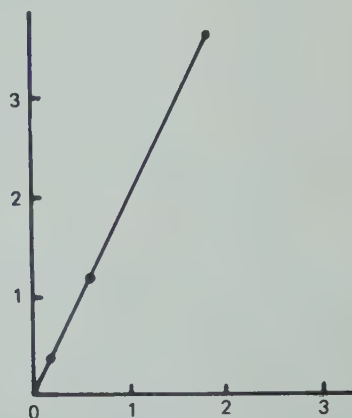
$$x = \frac{3}{y}$$

6	0.5
0.5	6
1.5	2
0.75	4



3) $x = ky$

$$k = \frac{x}{y} = \frac{0.2}{0.4} = \frac{1}{2}$$



4) a. $PV = k$

b. $k = 1 \text{ atm} \times 22.4 \text{ liters} = 22.4 \text{ liter-atm}$

c. $V = \frac{22.4 \text{ liter-atm}}{12 \text{ atm}} = 1.9 \text{ liters}$

$$V = \frac{22.4}{\frac{1}{2}} = 44.8 \text{ liters}$$

Appendix 7

Propagation of Errors in Calculated Results Addition and Subtraction

EXERCISES

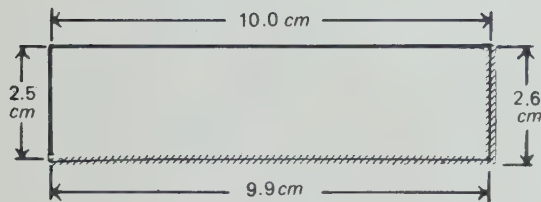
- 1) $30.0 \pm 0.4^\circ\text{C}$
- 2) $3962 \pm 22 \text{ cm}$
- 3) $5.11 \pm 0.02 \text{ g}$
- 4) $6.94 \pm 0.04 \text{ g}$

Multiplication and Division

- 5) Area of rectangle:
% uncertainty

$$(10.0 \text{ cm} \pm 1\%)(2.5 \text{ cm} \pm 4\%) \\ = 25.0 \text{ cm}^2 \pm 5\% = 25.0 \pm 1.2 \text{ cm}^2$$

Significant figures



$$(10.0 \text{ cm})(2.5 \text{ cm}) = 25 \text{ cm}^2$$

Area of shaded part

$$\begin{aligned} \text{at bottom } (9.9 \text{ cm})(0.2 \text{ cm}) &= 1.98 \text{ cm}^2 \\ \text{at right } (2.6 \text{ cm})(0.2 \text{ cm}) &= 0.52 \text{ cm}^2 \\ \hline &2.50 \text{ cm}^2 \end{aligned}$$

This answer is in rough agreement with $\pm 1.2 \text{ cm}^2$ and with $\pm 1 \text{ cm}^2$, the implied uncertainty in 25 cm^2 by the significant figure method. The use of highest and lowest value yields the same answer as % uncertainty.

$$2.6 \times 10.1 = 26.26 \text{ cm}^2$$

$$2.4 \times 9.9 = 23.76$$

$$\text{As found before: } 25.0 \text{ cm}^2$$

- 6) $200.0 \text{ g} \pm 0.5 \text{ g}$ is $200.0 \text{ g} \pm 0.25\%$
 $5.0^\circ\text{C} \pm 0.2^\circ\text{C}$ is $5.0^\circ\text{C} \pm 4\%$
 $(200.0 \text{ g} \pm 0.25\%)(5.0^\circ\text{C} \pm 4\%)$
 $\times 1 \text{ calorie/degree/gram}$
 $= 1000 \text{ calories} \pm 4.25\%$
 $= 1000 \pm 40 \text{ calories}$

Using significant figures, the least well known number has two (5.0°C) so the answer should have two, giving 1.0×10^3 .

$$7) \frac{9.8 \times 10^3 \text{ cal}}{1.23 \text{ g}} = 7.967 = 8.0 \times 10^3 \text{ cal/g}$$

(Note: Two significant figures.)

$$8) \frac{2.1 \times 10^2 \text{ cal}}{10.3 \text{ g}} = 20.4 = 2.0 \times 10 \text{ cal/g}$$

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NAME	SYMBOL	ATOMIC NUMBER	MOLAR MASS	NAME	SYMBOL	ATOMIC NUMBER	MOLAR MASS
Actinium	Ac	89	(227)	Mercury	Hg	80	200.6
Aluminum	Al	13	27.0	Molybdenum	Mo	42	95.9
Americium	Am	95	(243)	Neodymium	Nd	60	144.2
Antimony	Sb	51	121.8	Neon	Ne	10	20.2
Argon	Ar	18	39.9	Neptunium	Np	93	(237)
Arsenic	As	33	74.9	Nickel	Ni	28	58.7
Astatine	At	85	(210)	Niobium	Nb	41	92.9
Barium	Ba	56	137.3	Nitrogen	N	7	14.01
Berkelium	Bk	97	(247)	Nobelium	No	102	(253)
Beryllium	Be	4	9.01	Osmium	Os	76	190.2
Bismuth	Bi	83	209.0	Oxygen	O	8	16.00
Boron	B	5	10.8	Palladium	Pd	46	106.4
Bromine	Br	35	79.9	Phosphorus	P	15	31.0
Cadmium	Cd	48	112.4	Platinum	Pt	78	195.1
Calcium	Ca	20	40.1	Plutonium	Pu	94	(242)
Californium	Cf	98	(251)	Polonium	Po	84	210
Carbon	C	6	12.01	Potassium	K	19	39.1
Cerium	Ce	58	140.1	Praseodymium	Pr	59	140.9
Cesium	Cs	55	132.9	Promethium	Pm	61	(145)
Chlorine	Cl	17	35.5	Protactinium	Pa	91	(231)
Chromium	Cr	24	52.0	Radium	Ra	88	(226)
Cobalt	Co	27	58.9	Radon	Rn	86	(222)
Copper	Cu	29	63.5	Rhenium	Re	75	186.2
Curium	Cm	96	(248)	Rhodium	Rh	45	102.9
Dysprosium	Dy	66	162.5	Rubidium	Rb	37	85.5
Einsteinium	Es	99	(254)	Ruthenium	Ru	44	101.1
Erbium	Er	68	167.3	Samarium	Sm	62	150.4
Europium	Eu	63	152.0	Scandium	Sc	21	45.0
Fermium	Fm	100	(253)	Selenium	Se	34	79.0
Fluorine	F	9	19.0	Silicon	Si	14	28.1
Francium	Fr	87	(223)	Silver	Ag	47	107.9
Gadolinium	Gd	64	157.2	Sodium	Na	11	23.0
Gallium	Ga	31	69.7	Strontium	Sr	38	87.6
Germanium	Ge	32	72.6	Sulfur	S	16	32.1
Gold	Au	79	197.0	Tantalum	Ta	73	180.9
Hafnium	Hf	72	178.5	Technetium	Tc	43	(99)
Helium	He	2	4.00	Tellurium	Te	52	127.6
Holmium	Ho	67	164.9	Terbium	Tb	65	158.9
Hydrogen	H	1	1.008	Thallium	Tl	81	204.4
Indium	In	49	114.8	Thorium	Th	90	232.0
Iodine	I	53	126.9	Thulium	Tm	69	168.9
Iridium	Ir	77	192.2	Tin	Sn	50	118.7
Iron	Fe	26	55.8	Titanium	Ti	22	47.9
Krypton	Kr	36	83.8	Tungsten	W	74	183.9
Lanthanum	La	57	138.9	Uranium	U	92	238.0
Lawrencium	Lw	103	(259)	Vanadium	V	23	50.9
Lead	Pb	82	207.2	Xenon	Xe	54	131.3
Lithium	Li	3	6.94	Ytterbium	Yb	70	173.0
Lutetium	Lu	71	175.0	Yttrium	Y	39	88.9
Magnesium	Mg	12	24.3	Zinc	Zn	30	65.4
Manganese	Mn	25	54.9	Zirconium	Zr	40	91.2
Mendelevium	Md	101	(256)	(unnamed)*	?	104	(260)

* Discovery reported in 1964.

Molar masses based on $^{12}\text{C} + 12.000$.

Numbers in parentheses give the mass number of the most stable isotope.

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